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Journal

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American Ceramic Society

A Monthly Journal devoted to the Arts and Sciences Related to the Silicate Industries.

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A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **May, 1919** **No. 5**

EDITORIALS.

THE SUMMER MEETING.

As previously announced in these columns, the 1919 Summer Meeting of the American Ceramic Society will be held at Buffalo, Niagara Falls and Cleveland, August 4th to 8th, inclusive, the meeting to convene at the Hotel Statler, Buffalo, on Monday evening, August 4th. As usual the daytime will be spent in inspection trips to plants of ceramic interest in the vicinity of the above cities—the evenings being devoted to such business meetings and entertainments as are being arranged by the Committee on Summer Meeting of the Society and the local entertainment committees.

The Refractories Division announces a meeting to be held at Niagara Falls on August 4th, at which several important matters dealing with the future work of the Division will be discussed. Meetings of other Divisions will be held during the week. Cooperating with the Summer Meeting Committee, the Northern Ohio Section is arranging an itinerary and entertainment for the visiting members of the Society while in Cleveland and vicinity.

The central location, excellent rail and boat facilities, and the many ceramic plants of interest in the Buffalo and Cleveland districts should assure an excellent attendance at this meeting. Aside from the opportunity to visit the modern pottery, glass and enameling plants at Buffalo and Cleveland, the electrochemical plants at Niagara Falls should prove of unusual interest to our members.

Full details of the arrangements for the meeting will be announced in the June number of the Journal.

PROFESSIONAL DIVISIONS.

It is gratifying to note that the Terra Cotta Division of the American Ceramic Society is now fully organized and that considerable progress has been made in outlining and getting under way the work for this year. Enrolled in this Division are forty-nine members representing twenty-four terra cotta companies, the Bureau of Standards, and the Ohio State University.

Both the Ohio State University and the Bureau of Standards are now doing research work on terra cotta bodies and are willing at all times to coöperate with members of the Division in solving any of their problems and in conducting the investigations which have been undertaken by the members of the Division in their own laboratories and plants. These investigations cover a wide field including: the improvement and development of glazes; the effect of electrolytes on terra cotta bodies; the effect of the size of grog in terra cotta bodies; spalling of terra cotta bodies; spraying machines; humidity drying; firing terra cotta kilns; standardization of terra cotta colors; etc.

It is the intention of the officers of the Division to have the results of as many of the above investigations as possible prepared for submission to the members of the Division some time before the next annual meeting of the Society, in order to allow a free discussion before their formal presentation before the Society.

The terra cotta industry should be well pleased with the progress made in the last few months in technical coöperation between the companies and the American Ceramic Society and with the interest shown in getting some valuable research work started which is bound to result in improved methods of manufacture, better terra cotta and fewer failures. The formation of the Terra Cotta Division at the last annual meeting has already been more than justified.

When the rapidly maturing plans and investigations of the other Divisions of the Society are well under way, there will be no question as to the value of group organizations of this kind in the technical advancement of the different branches of the ceramic industry and in adding to the technical literature and membership of the Society.

AMERICAN ENGINEERING STANDARDS ASSOCIATION.

The American Engineering Standards Committee has just completed a revision of its Constitution which has been sent to the Governing Boards of all the Departments and Societies represented on the Committee with a request for its ratification. The new Constitution changes the name to American Engineering Standards Association, the change from "Committee" to "Association" more fittingly indicating the wide scope of the interests involved.

Included in the Founder Societies of the Committee were the American Society of Civil Engineers, American Institute of Mining Engineers, American Society of Mechanical Engineers, American Institute of Electrical Engineers and the American Society for Testing Materials. The objects of the Association are stated as follows:

1. To unify and simplify the methods of arriving at engineering standards, to secure coöperation between various organizations and to prevent duplication of standardization work.
2. To promulgate rules for the development and adoption of standards.
3. To revise and pass upon recommendations for standards submitted as provided in the Rules of Procedure, but not to initiate, define or develop the details of any particular standard.
4. To act as a means of intercommunication between organizations and individuals interested in the problems of standardization.
5. To give an international status to approved American engineering standards.
6. To coöperate with similar organizations in other countries and to promote international standardization.

In view of the importance of standardization in the testing and classifying of ceramic materials and products and of the extensive work now being carried on by the Committee on Standards of the American Ceramic Society, it would appear that the Society should be vitally interested in furthering the purposes of the Standards Association. In the past there have been duplications and wasted efforts in attempts to standardize tests for ceramic materials. There should be technical coöperation without as well as within the Society in arriving at suitable standards.

ORIGINAL PAPERS AND DISCUSSIONS

A STUDY OF SOME LIGHT-WEIGHT CLAY REFRACTORIES.

By M. F. Beecher.

The work described in this paper was suggested by the investigation on light-weight refractories promoted by the Pittsburgh Laboratory of the Bureau of Standards and conducted by them in coöperation with the Navy Department. Their development of a brick of attractive quality has been the incentive inspiring others to a similar line of work, and enough interest is being taken in this new product to indicate that it may have a considerable field of usefulness.

It is not the purpose here to bring out the advantages or properties of specific composition, so much as to show by means of the data presented some of the factors involved and some of the limits met in the making of light-weight clay refractories. The data are not sufficient, either in volume or detail, to cover the subject thoroughly, but the comparative nature of most of the results will indicate the general quality of brick which may be produced.

The Bricks Tested.—In this study, bricks of 12 different compositions were subjected to test. The first seven of these—those upon which our attention will be particularly centered—were experimental light-weight bricks. The other five were full-weight commercial bricks tested to supply a basis for comparison. The compositions appear in Table 1, the parts being by volume.

TABLE 1.—COMPOSITIONS OF THE BRICKS TESTED.

No.	Lab. No.	No. 1 refractory clay. Per cent.	Kaolin. Per cent.	Regular grog. Per cent.	Kaolin grog. Per cent.	Sawdust. Per cent.
1....	M84	19	38	24	..	19
2....	M86	10	30	13 1/3	..	46 2/3
3....	M87	50	10	....	10	30
4....	M88	50	10	10	..	30
5....	M89	50	10	20	..	20
6....	M91	50	20	....	..	30
7....	M94	25 1/3	24 2/3	....	..	50
8....	M77	Norton special clay brick.			..	..
9....	M22	A good quality silicious clay brick made in New England of New Jersey clays.				
10....	M85	A Pennsylvania flint-clay brick.				
11....	M90	A Missouri flint-clay brick.				
12....	M95	A Kentucky flint-clay brick.				

Each of the last four commercial fire bricks described above represents approximately the best quality produced in each district. The grog referred to as regular grog is that which is produced in reclaiming broken slabs and saggers. It contains, of course, no slag or fusible material of any kind. The kaolin grog was prepared by calcining washed Florida kaolin at cone 13. In mixtures of this kind the greatest variation is likely to be found in the kind or character of sawdust used. The sawdust in these bricks contained no hard woods. It had a weight of 14 pounds per cubic foot when shaken down slightly and was sized to pass an 8-mesh screen.

The bricks were made by hand in single wooden molds from a mixture thoroughly pugged in a small auger machine. The manufacturing data appear in Table 2 following:

TABLE 2.—MANUFACTURING DATA.

No.	Lab. No.	Workability.	Drying behavior.	Drying shrinkage.	Burning shrinkage.	Total shrinkage.
1....	M84	Sl. short	Good	4.7	4.6	9.1
2....	M86	Short	Good	4.4	7.8	11.4
3....	M87	Good	Good	4.0	3.8	7.6
4....	M88	Good	Good	4.8	3.2	7.8
5....	M89	Good	Good	4.0	3.2	7.1
6....	M91	Sl. short	Good	5.1	3.7	8.6
7....	M94	Short	Good	5.1	7.2	11.9

Cold Physical Properties.—A more complete description of the bricks is provided by the data of Tables 3 and 4 which follow. The cold physical properties given in Table 3 were obtained by the usual methods. They consisted of the determination of the modulus of rupture, porosity, apparent specific gravity, bulk specific gravity, and the weight reduced to the standard $2^1/_2'' \times 4^1/_2'' \times 9''$ size.

TABLE 3.—COLD PHYSICAL PROPERTIES OF THE BRICKS TESTED.

No.	Lab. No.	Modulus of rupture, lbs. per sq. in.	Per cent porosity.	Bulk sp. gr.	Apparent sp. gr.	Wt. per std. $2\frac{1}{2}'' \times 4\frac{1}{2}'' \times 9''$, pounds.
1.....	M84	716.0	39.5	1.54	2.57	5.51
2.....	M86	270.0	56.5	1.11	2.59	4.01
3.....	M87	547.0	47.7	1.46	2.80	5.43
4.....	M88	474.0	46.1	1.50	2.80	5.50
5.....	M89	436.0	43.0	1.60	2.81	5.84
6.....	M91	533.0	51.8	1.48	3.06	5.35
7.....	M94	283.0	60.6	1.10	2.81	3.93
8.....	M77	558.0	31.3	1.87	2.72	6.76
9.....	M22	304.0	32.0	1.82	2.65	6.58
10.....	M85	436.0	21.0	1.94	2.46	6.87
11.....	M90	...	17.4	2.17	2.63	7.92
12.....	M95	457.0	22.1	2.09	2.68	7.43

The modulus of rupture was calculated by the usual formula $R = \frac{3\ Pl}{2\ bd^2}$ from the transverse breaking load on five specimens. The breaking machine was of simple construction, having a lever ratio of 1 to 20. The knife edges were of steel and rounded to a radius of $^1/_4''$. One was fixed and the other two free to move, thus allowing for any unparallel condition of the specimens. The load was applied by means of falling shot and the machine load measured was $^1/_2$ P.

The porosities were determined upon half-bricks resulting from the transverse breaking test. The dry weights, saturated weights and suspended weights were obtained using distilled water as the saturating and suspending medium. Saturation was accomplished by boiling submerged for one hour and the calculation was by Purdy's formula $\frac{W - D}{W - S} \times 100 =$ per cent porosity.

The specific gravities were obtained from the same values by the relations $\frac{D}{D-S}$ = apparent specific gravity, and $\frac{D}{W-S}$ = bulk specific gravity.

The weights reduced to standard dimensions were obtained by calculations from actual measurements and weights.

TABLE 4.—AVERAGE WEIGHTS OF COMMERCIAL FIRE BRICKS.

Source.	Number of brands.	Maximum weight, pounds.	Minimum weight, pounds.	Average weight, pounds.
Pennsylvania.....	11	8.22	6.97	7.46
W. Virginia.....	1	..	..	7.58
Illinois.....	2	7.42	6.89	7.15
Maryland.....	6	8.11	7.05	7.34
Missouri.....	7	7.81	7.22	7.56
Kentucky.....	2	8.55	8.14	8.34
Colorado.....	1	..	..	7.44
Ohio.....	7	7.68	6.56	7.14

Table 4 above presents some data on the weights of commercial fire brick which will be of interest in connection with the data of Table 3. Of the 37 different brands of commercial fire brick represented here the average weight was found to be 7.46 pounds. In general, there appears to be a very wide variation in the weights of fire bricks from the same locality; too much importance therefore should not be placed upon these averages resulting from a very small number of bricks—for instance, the average of 8.34 pounds for Kentucky brick, where only two different brands were available, is hardly representative.

Chemical Analyses.—In Table 5 appear the chemical analyses of these bricks. In several cases the mixtures differed only in the quantity of sawdust present and only one analysis, therefore, was made to cover such cases.

It is not expected that any deduction as to the quality of these bricks can be made from these analyses, but they will show what wide variations in chemical composition are possible between bricks of equal or similar ultimate quality.

TABLE 5.—CHEMICAL ANALYSES OF THE BRICKS TESTED.

No.	Lab. No.	SiO_2.	Al_2O_3.	Fe_2O_3.	TiO_2.	CaO.	MgO.	K_2O and Na_2O.	Total fluxes.
1	M84	...	...	..	..	..	..	..	..
2	M86	62.66	33.96	0.46	1.70	0.54	0.62	0.28	1.90
3	M87	...	...	..	..	..	..	..	..
4	M88	41.84	52.77	1.11	3.10	0.32	0.70	0.40	2.53
5	M89	44.52	50.31	0.51	3.50	none	0.73	0.30	1.54
6	M91	28.08	64.21	2.91	4.10	none	0.67	0.28	3.86
7	M94	...	...	..	..	..	..	..	..
8	M77	45.64	48.09	2.07	2.20	0.56	0.55	0.56	3.74
9	M22	80.02	13.86	2.72	1.70	0.36	0.63	0.62	4.33
10	M85	50.24	43.18	1.94	3.00	0.44	0.81	0.32	3.51
11	M90	57.14	37.55	1.83	1.60	0.56	0.77	0.54	3.70
12	M95	58.64	35.22	2.64	2.50	0.08	0.77	0.22	3.71

Load Carrying Capacity at Elevated Temperatures.—The load test method conformed to the A. S. T. M. tentative standard.[1] A load of 50 pounds per square inch was used because a considerable amount of data obtained from tests under those conditions was already available for comparison, and also because the greater load would allow of greater deformation thus serving to differentiate more closely between the bricks of highest quality. In Table 6 following are presented the data resulting from this test:

A discussion of these results[2] has brought out that in comparison with tests on the same brands of commercial brick in another laboratory, the high deformation here noted might indicate that the test conditions applied were somewhat severe. However, very close attention was given to the set-up and manipulation. The thermocouple cold-junction was maintained between 20° and 25° C throughout the test. It was learned later[3] that the commercial brick represented by No. 12 did not appear to be as dense or as well burned as those ordinarily produced by this manufacturer. We, therefore, can account for this apparent variance of results in the possible difference in quality of bricks of the same brand[4] and the possible use of different types of testing

[1] *Proc. A. S. T. M.*, **17**, Part 1, p. 665 (1917).

[2] Private correspondence.

[3] Information from an extensive user of that brand.

[4] *J. Am. Ceram. Soc.*, Vol. 1, p. 403. Loomis mentions this possibility.

TABLE 6.—RESULTS OF LOAD TESTS.

50 pounds per sq. in. at 1350° C.

No.	Lab. No.	Initial length, inches.	Final length, inches.	Deformation.		Remarks.
				Inches.	Per cent.	
1...	M 84	9.03	8.79	0.24	2.66	.....
2...	M 86	8.80	7.87	0.93	10.57	.....
3...	M 87	9.25	Shear	..	...	45 minutes at 1350° C.
4...	M 88	9.19	8.68	0.51	5.54	.....
5...	M 89	9.28	9.06	0.22	2.37	.....
6...	M 91	9.21	8.79	0.42	4.56	.....
7...	M 94	8.85	6.74	2.11	23.80	30 minutes at 1350° C.
8...	M 77	9.07	8.84	0.23	2.54	.....
8...	M 77	9.08	8.91	0.17	1.87	.....
9...	M 22	9.01	8.88	0.13	1.33	.....
10...	M 85	9.22	8.57	0.65	7.06	.....
11...	M 90	8.78	8.04	0.74	8.43	.....
12...	M 95	8.86	7.23	1.63	18.40	.....

furnaces in the two laboratories. In any event it is evident that the results presented in Table 6 are comparable within this study.

Fig. 1 shows the manner off ailure of the bricks tested. The photographs exhibit an interesting feature, namely, the absence of serious buckling of the specimens. Even where the deformation is very great, as in Nos. 7 and 12, the buckling was not sufficient to have been a serious factor in the deformation.

Spalling Behavior.—The spalling test followed somewhat the method of Nesbitt and Bell,[1] except that the cooling was accomplished in air instead of in water. The heating was done in the standard load-test furnace. The heating chamber was filled with a checker-work of fire brick, set on edge, built up two high. The thermocouple hot-junction was within 1″ of the lower end of the test bricks and approximately in the center of the furnace. Since the capacity of the furnace was eight bricks, two runs were necessary, but care was taken to see that the same rate of heating and the same cooling intervals were followed in both runs.

The furnace as described, with the test bricks in place, was heated up to approximately 1300° C and the bricks then with-

[1] *Proc. A. S. T. M.*, **16**, part 2, p. 349.

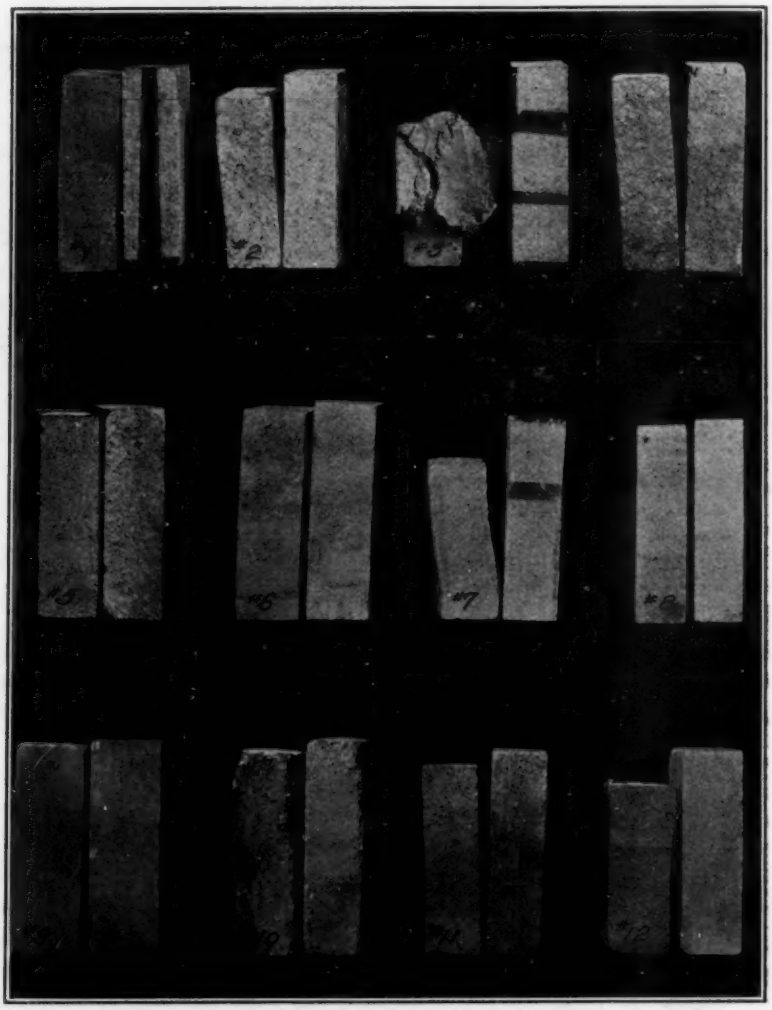

FIG. 1—Behavior of bricks in load test.

drawn one at a time and cooled in a blast of air for exactly one minute. The air issued from a nozzle, $^5/_8''$ in diameter and $4''$ from the bricks, under a pressure of five pounds. The blast was directed at right angles against the hot end. This was repeated

every 20 minutes—since that was the time necessary for the furnace temperature to return to approximately 1300° C. The test was continued to 30 heatings and coolings.

The operation of removing from the furnace and returning the cooled brick to the furnace required on the average of 15 seconds. During the first ten minutes, therefore, of the 20 minute cycle there were only seven bricks continuously in the furnace and the temperature would drop from 1300° to 1250° C. During the last ten minutes of the cycle the eight bricks would be in place and the temperature would return to 1300° C again by the next cooling period. Notes on the development of cracks and spalls were taken during the test—for many cracks that were very apparent while the brick was under the air blast could not be distinguished when the brick had finally cooled.

In Fig. 2 are shown, graphically, the results obtained. While Nesbitt and Bell[1] have measured the spalling by the loss in weight of the bricks at the conclusion of thirty coolings, this did not appear to afford a good means of judging the results in the present instance. Many of the bricks showed no loss in weight whatever but these could be quite readily classified on the basis of the extent of cracking. Thus we have indicated as accurately as possible the extent and position of the cracks in one face and in the heated end of each brick, after the thirtieth cooling, or the discontinuance of the test. The double line indicates a complete fracture or spall while the single line represents only a crack. The small number in the upper right hand corner of each brick indicates its rank as determined by this test, brick marked "1" showing the greatest resistance to spalling.

Nor is this method of classification found entirely satisfactory. No. 2 and No. 7 have been ranked 12 and 11, respectively, while it is entirely probable that the fractures produced during the test were not due to the repeated heating and cooling as much as to the rather rough handling which they of necessity received. These two bricks, it will be observed upon reference to Table 3, have very low mechanical strength and the effect of the handling would naturally be exaggerated in proportion. Brick No. 3

[1] *Loc. cit.*

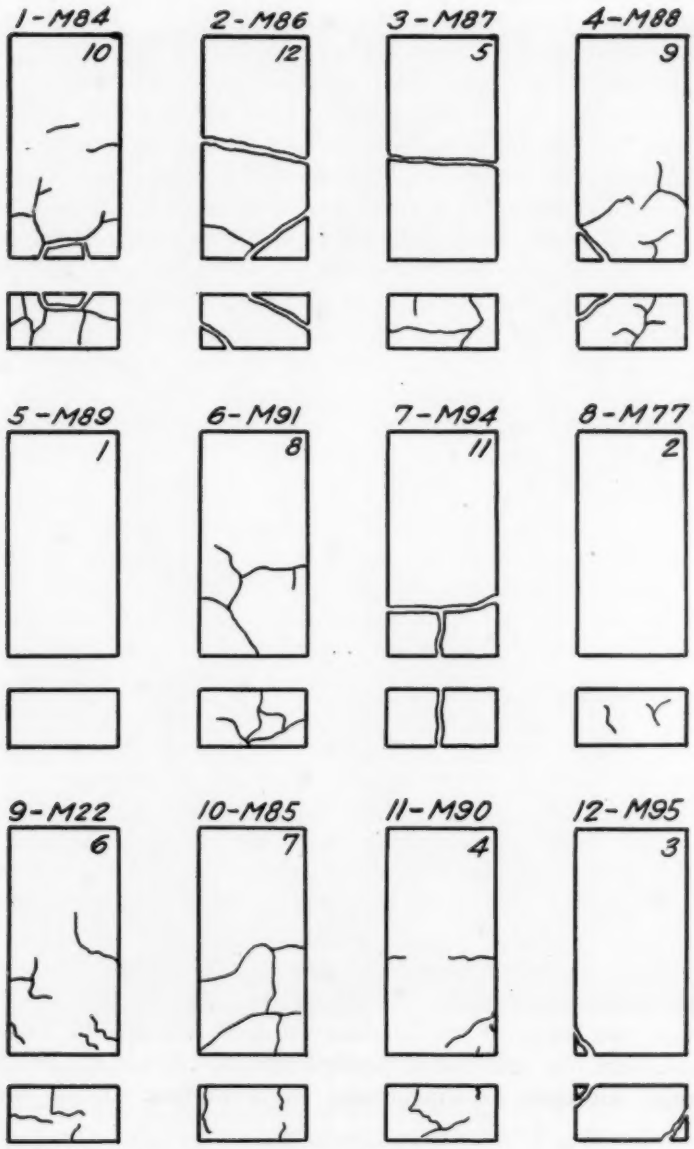

FIG. 2.—Results of spalling tests.

showed very few cracks after the test, but on removing from the furnace after the thirtieth heating, it was found to be in two pieces. This case also, we are satisfied, can be attributed to the low mechanical strength of this mixture.

Fusion Point.—The fusion point determinations were made through the courtesy of Mr. R. H. White.[1] The test cones were made by crushing portions of each of two bricks, amounting to approximately one-quarter of a brick, between steel surfaces to pass a 60-mesh screen. An addition of gum was then made and cones of the standard dimensions molded in a brass mold. These were set up with standard cones on refractory pats made of a mixture consisting of 85 per cent of fused alumina and 15 per cent of refractory bond clay. The cone pats were $2^1/_4''$ wide and $5''$ long, and each contained two rows of cones of seven cones each. In each row the four standard cones were alternated with three test cones. The cones in the two rows were placed in reverse order, thus preventing any unequal heating of the furnace from affecting the results.

In Table 7 are given the results obtained in the fusion tests:

TABLE 7.—RESULTS OF FUSION TESTS.

Number.	Laboratory number.	Cone of fusion.
1	M84	31–32
2	M86	31–32
3	M87	31–32
4	M88	31–32
5	M89	31–32
6	M91	32
7	M94	32
8	M77	32
9	M22	29–30
10	M85	32
11	M90	31–32
12	M95	Not made

Thermal Insulation.—Only a simple and approximate test was made to determine the relative behavior of the bricks as regards

[1] Tech. Director, Abrasive Plants, Norton Company, Niagara Falls, N. Y.

thermal insulation. It is not intended that the results presented should be considered as accurate or even very exact measures of the insulation properties. The factors involved in an exact thermal study are so many and varied and the importance of very careful manipulation so great that there was neither the time, nor in fact, the necessity for it in the present instance. However, the information conveyed in the curves of Fig. 3 shows roughly the relative behavior of the bricks as insulators.

These data were obtained by subjecting one face of the test specimen to the direct flame of a gas burner and noting the temperature rise per time of the opposite face. The temperature of the flame was constant at 1100° C ± 15°. The set-up was exactly the same for every test and was protected against variable air currents. Readings were taken at five-minute intervals for one hour or until the cooler face reached a temperature of 350° C.

For purposes of comparison, a Silocel insulating brick and a Crystolon brick were subjected to the same test and the curves plotted as for the others.

While the differences between the bricks are not especially marked, they are sufficient to differentiate roughly between the good and the poor The experimental clay bricks average up equal or superior to all the commercial brands except No. 12.

Summary.

For convenience the major results from each of the preceding tests are assembled in Table 8. These show that, except for Nos. 2 and 7, the load-carrying capacities compare very favorably with the standard full-weight bricks. In resistance to spalling the light-weight bricks again show up very well. In thermal insulation they are practically equal to or superior to all the commercial brands except No. 12.

The weights of the experimental bricks are somewhat parallel with the porosities, though the character of the clays used may have a marked influence on weight.

The porosity is governed by the character of the clays, the amount and size of the grog, and the amount of sawdust added. The more open-burning clays and the more uniformly sized grog

TABLE 8.—SUMMARY OF DATA FROM COMPARATIVE TESTS.

No.	Weight.		Load Test.		Resistance to spalling.		Cone of fusion.	Thermal insulation. Rank.
	Rank.	Standard $2\frac{1}{2}'' \times 4\frac{1}{2}'' \times 9''$.	Rank.	Per cent deformation.	Rank.	Behavior.		
1..	6	5.51	4	2.66	10	fair	31–32	10
2..	2	4.01	9	10.57	12	poor	31–32	3
3..	4	5.43	12	Shear	5	good	31–32	5
4..	5	5.50	6	5.54	9	fair	31–32	6
5..	7	5.84	3	2.37	1	good	31–32	2
6..	3	5.35	5	4.56	8	fair	32	4
7..	1	3.93	11	23.80	11	poor	32	8
8..	9	6.76	2	2.19	2	good	32	11
9..	8	6.58	1	1.33	6	fair	29–30	9
10..	10	6.87	7	7.06	7	fair	32	7
11..	12	7.92	8	8.43	4	good	31–32	12
12..	11	7.43	10	18.40	3	good	...	1

will naturally make the more porous brick. Sawdust additions (to relatively open-burning clay mixtures) appear to increase the porosity by an amount equal to the actual volume of the addition. Thus, comparing M77 and M88, which are very similar in clay content, we find the porosities to be 31.3 per cent and 46.1 per cent with sawdust contents of 0 and 30 per cent, respectively. The actual volume of this sawdust is about one-half its bulk volume, or, the wood particles added actually constitute but 15 per cent. The difference in the porosities is 14.8 per cent. M91 and M94 have porosities of 51.8 per cent and 60.6 per cent with a difference in sawdust content of 20 per cent. The difference in porosity is 8.8 per cent.

The *load carrying capacity* of fire bricks at elevated temperatures, it has been observed, seems to have no relation to the strength in the cold condition.[1] When the compositions are similar, however, and the porosity is varied by induced methods, the deformation varies with the porosity or openness. Examples are those of M87, M88 and M89.

The results obtained on the light-weight bricks show that all

[1] A. V. Bleininger and G. H. Brown, Bur. of Stds., *Tech. Paper*, 7, p. 61.

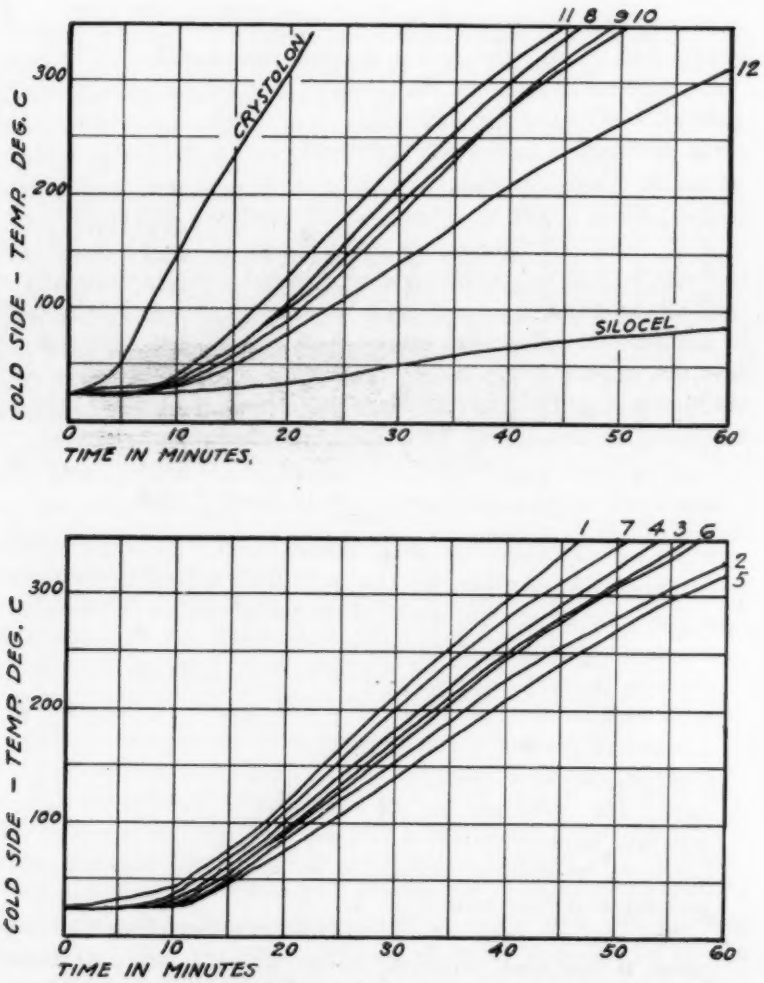

FIG. 3.—Relative insulation of 2½" brick. Temperature of hot side, 1100° C. ± 15° C.

but No. 3 (M87) and No. 7 (M94) deformed less than the poorest commercial brand. Four showed lower deformations than three of the five commercial bricks.

The *spalling test* results seem to bear no relation whatever to the bulk specific gravity, the porosity, the deformation under

load, or the cold strength. There is, however, quite a marked relation between spalling behavior and thermal insulation, as may be observed by reference to Figs. 2 and 3. This is especially interesting, notwithstanding the fact that the object of each of these tests was to obtain only comparative values for the different bricks. It presents an interesting topic for further study.

The character of the clays constituting the several bricks seems to have little influence on the spalling behavior. In the present study, however, the variety of clays used is too limited to warrant a general statement. The physical properties of the bricks seem to be the factors of real influence, though the manner in which each operates is not disclosed by the present set of data.

The *thermal insulation* data cannot be readily correlated with the compositions, for the field of compositions covered is too small. However, there are several interesting features to be pointed out. Contrary to our usual conception there is no relation, in the present case, between porosity and insulation property. In mixtures M91 and M94, the more porous brick (M94) is the poorer insulator. The same fact appears as well in M87, M88 and M89.

It should be noted that all of the experimental bricks are grouped within the extreme limits set by the commercial brands.

Conclusions.

1. The working properties and drying behavior of a fire clay mixture are affected by additions of sawdust in the same manner as by additions of grog.

2. Between 15 and 20 per cent of sawdust may be added to mixtures of the type studied without appreciably affecting the total shrinkage.

3. Raw kaolin increases the drying and burning shrinkages, but may be used without harm up to approximately one-third of the plastic clay content.

4. Not more than 45 per cent of sawdust can be added and still maintain a workable mixture. Forty per cent is about the practical limit.

5. At least 45 per cent of plastic clay (not more than 15 per cent kaolin) is necessary for proper working qualities.

6. The weight of the bricks is controlled largely by the amount of combustible material added, but the porosity and apparent specific gravity of the clay used has sufficient influence to be of importance.

7. In open-burning mixtures, additions of sawdust increase the porosity of the burned bricks by an amount equal to approximately one-half of the bulk volume added.

8. There appears to be some relation between the tendency to spall and the property of thermal insulation of the bricks tested in this study. The bricks of the better insulating qualities seem to spall the least.

9. There seems to be no relation between porosity and thermal insulation of these bricks, contrary to what would naturally be expected.

10. This study would indicate that approximately four pounds is the minimum weight that can be reached in a light-weight brick to satisfactorily compete against standard No. 1 fire brick and still retain sufficiently good working properties in the mixture to make commercial manufacture possible.

11. The load test appears to offer a better and surer means of judging these bricks than any other, for the light-weight bricks compare favorably with the commercial bricks in all tests except this.

12. In general the data presented offer some encouragement and indicate the possibility of producing, commercially, very satisfactory fire brick of very low weight.

RESEARCH LABORATORIES,
NORTON COMPANY
WORCESTER, MASS.

COMMUNICATED DISCUSSIONS.

D. W. Ross: In light-weight clay refractories for furnace linings, we must expect to sacrifice something in heat insulating value, for of necessity the weight of a brick depends largely upon its porosity.

This is in accordance with the results of the work of Ray and Kreisinger,[1] who found that in furnace walls fire bricks give

[1] W. T. Ray and Henry Kreisinger, U. S. Bur. Mines, *Bull.* 8 (1911).

greater insulation than a dead-air space, and who state that "when heat at low temperature is insulated, use air space; when the heat is at high temperature, as is the case in furnaces, use solids of poor conductivity."

In general, Kreisinger found that a 2-inch brick wall was more advantageous than a dead-air space above 352° C, and a 4-inch brick wall was more advantageous than a dead-air space above 227° C—when the measurements were based on a drop in temperature of 100° C. This in turn is in accordance with the laws of radiation and conduction. We know from the Stefan-Boltzmann law that radiation increases approximately as the fourth power of the difference in temperature between the hot and cold body, while the "amount of heat conducted through a unit of area from one part of a body to another is proportional to the temperature difference of the two parts; is proportional to the conductivity of the body; and is inversely proportional to the distance between the two parts of the body." Hence, as stated above, at furnace temperatures we would expect more space to decrease the insulating power of a brick to some extent, instead of increasing it.

With reference to the rate of cooling of clay fire-brick, above dull red heat, we know that silica and clay probably have slightly different thermal capacities and thermal conductivities, and that conductivity may be retarded in a brick by lack of contact of adjoining particles, due to porosity, or be otherwise affected by the degree of vitrification of the brick. We have a few data, however, that appear to indicate that the controlling factor in the cooling of clay fire-brick is the mass per unit of volume; that the rate of cooling (drop in temperature) decreases as the mass per unit volume increases, or, in this case, it is probably safe to say that in spite of differences in per cent of silica present, porosity and degree of vitrification, the controlling factor in the rate of cooling (drop in temperature) of a clay fire-brick, above dull red heat, is the actual quantity of heat stored in the brick.

Mr. Beecher's paper appears to the writer as an excellent contribution to the subject of light-weight clay refractories and is of special value because of the direct correlation of the light-weight

refractories with sufficient numbers of standard commercial brands of fire brick to give us a perspective of the relation of properties between the former and commercial fire brick.

At least where large percentages of kaolin have been used in the manufacture of light-weight refractories, the surface of the brick, where a gas or oil flame impinges directly upon it, soon shrinks and develops many cracks similar to those appearing on the surface of a sun-baked mud pond. Such high kaolin bricks, especially when very porous, are apt to contract (not necessarily soften and settle) in the load test. However, it is our opinion that with the right clays and methods of handling, a refractory brick of comparatively light weight can be produced that will meet gas and oil-fired furnace conditions and be well within the limits of the load test (1350° C and 50 pounds per square inch).

LABORATORY
THE FINDLAY CLAY POT CO.,
WASHINGTON, PA.

R. L. CLARE: Our interest in this class of refractories was caused by inquiries made by the Navy Department, both at the Brooklyn and Philadelphia Navy Yards, for a porous light-weight refractory having certain qualities. We submitted samples to the Philadelphia Navy Yard for testing and made the pieces for one installation from this type of material.

Ground cork (8–16 mesh) was used to secure the desired porosity. This was decided upon after numerous difficulties encountered in securing a uniform grade of sawdust. No two lots of sawdust were found alike, either in the kind of wood, or size and shape of the grains. Some of the grains would be more or less cubical, while others would be slivers. The cork as received eliminated all of these difficulties.

The balance of the mixture was made up of Florida kaolin, a plastic New Jersey fire clay and 4–16 mesh ground, fire-brick grog. Numerous mixtures were prepared, the most successful of which, when used for bricks only, was of the following proportions:

	Per cent.
8–16 mesh cork	49.2
Fire clay	15.7
Florida kaolin	22.2
4–16 mesh grog	12.9
	100.0

This mixture pugged easily, pressed well, and dried satisfactorily. It is very important, especially if shapes other than brick are made, that a safe drying mixture be obtained. The properties of this mixture when burned to Cone 14 were as follows:

Total shrinkage	10.3%
Absorption	50.0%
Bulk sp. gr.	1.1
Weight of $9'' \times 4^1/_2'' \times 2^1/_2''$ brick	4.0 pounds

This material should easily stand cone 31, although no softening point determinations were made. We know, however, that all the inorganic materials which entered into the mixture have softening points above cone 31. Load tests were not made, as no means were at hand within the time available, but such a test should be made. In comparing the properties of the above brick with those found by the author, it would seem that the porosity of this mixture is a little too high when used under load conditions under fire.

Apparently it is possible to add a higher percentage of ground cork than of sawdust to a mixture of this type, as we used 50 per cent cork and still maintained safe working properties, whereas the author maintains that only 40 per cent sawdust can be added safely.

In making slabs or other shapes larger than a regular brick, the warpage of the mixture needs serious consideration. The porosity and shrinkage must be materially reduced over that shown above, to be safe for shapes. The exact limits for pieces of stated sizes has not been determined.

FEDERAL TERRA COTTA CO.,
WOODBRIDGE, N. J.

R. M. HOWE: It is hardly possible to offer more than a favorable criticism to the above paper. However, it is questionable whether or not such a heavy load should be applied to the light-weight bricks. Because of their nature the use of such bricks would tend to greatly reduce the load effect and, out of fairness to them, load tests using pressures proportional to the weight of the bricks might well be considered.

Some interesting data is at hand which concerns the more silicious light-weight bricks. A $9'' \times 2^1/_2'' \times 4^1/_2''$ brick is at hand which weighs 814 grams. Its fusion point is about cone 31. Under the tentative standard load test for silica brick, but with a pressure of 20 pounds per square inch, similar bricks sheared after being held one-half hour at 1500° C.

MELLON INSTITUTE,
PITTSBURGH, PA.

F. H. RIDDLE: The results obtained by Mr. Beecher in his work on light-weight refractories are very interesting, especially as there is a distinct demand for this kind of product for marine boiler installations. The data presented is very complete and checks the results obtained in our laboratory. The fact that the thermal conductivity did not vary with the porosity is probably to be ascribed to the size of the pores. When these are large, transfer of heat by radiation from one side of a cavity to the other becomes a factor which is less prominent than in the case of a large number of fine pores.

It might be interesting to add that the first government order for a product of this kind was filled by the Harbison-Walker Refractories Company at Pittsburgh. The work done so far seems to show that there is a distinct commercial application for light-weight refractories.

BUREAU OF STANDARDS,
PITTSBURGH, PA.

M. F. BEECHER: The writer does not entirely agree with Mr. Ross that we must expect to sacrifice heat insulating value for increased lightness in weight, when the latter is obtained by in-

duced porosity. The conclusions drawn by Ray and Kreisinger[1] do not cover precisely the insulation problem these bricks present. Carl Hering has discussed their work[2] and has pointed out that the phenomenon of "contact resistance" is probably involved as well as that of radiation, and therefore a furnace wall will offer better insulation if the customary air space is filled with sand or a similar loose granular material. Likewise, a single brick extending through a furnace wall will conduct more heat, and be hotter on the outer end, than an adjacent brick which is not continuous through the wall but has several intervening cracks or joints. Induced porosity may have a somewhat similar effect upon the transfer of heat, and for similar reasons. It does not necessarily follow that because a material is more porous it should be a poorer insulator. When we think of heat insulators generally, we usually visualize a light, porous material such as Silocel, Nonpareil brick, or cork. It appears, however, that the size and character of the cells or pores, as Mr. Riddle points out, may be the factor that governs the effect of induced porosity upon heat conductivity.

Mr. Clare's contribution to this discussion is very interesting and valuable. There are great differences in sawdust from different sources and care must be exercised if a uniform quality is always to be obtained.

The silicious light-weight brick mentioned by Mr. Howe, which weighs 814 grams, corresponding to a weight of approximately two pounds in the standard size, is very interesting. A more extended description would be an interesting contribution to the subject of light-weight refractories.

[1] Bur. Mines, *Bull.* **8** (1911).

[2] *Met. and Chem. Eng.*, **9**, 438.

THE VOLATILIZATION OF IRON FROM OPTICAL GLASS POTS BY CHLORINE AT HIGH TEMPERATURES.

By J. C. Hostetter, H. S. Roberts, and J. B. Ferguson.

The presence of iron in optical glass arises from two main sources, namely, the raw materials and the pots used for melting.[1] Improvement in the purity of raw materials was established in advance of the development of good pots, with the result that for many months the amount of iron absorbed by the glass from the pot was several times that introduced from the batch. In the expectation of reducing the amount of iron contributed to the glass by the pots then available to an amount not greater than that introduced with the batch, the experiments described below on the volatilization of iron from pots by the action of chlorine were carried out. The experiments indicated beyond doubt that iron could be readily removed from pots by this method, which requires very simple equipment and can be applied during the "arching" or burning period; the cost of the process is negligibly small in comparison with other costs involved in making optical glass, and the technic required is simple. The large scale experiments to be described here were, however, only partially successful—not on account of deficiencies inherent in the method, for the iron was actually removed from the pot—but on account of other conditions. Inasmuch as there are many ceramic materials whose iron content can be decreased by this method, the details of this investigation are recorded here in order that our experience may be available to any others who are interested.

Iron Absorbed from Pots.—For illustrative purposes, Table 1 has been prepared in order to show the variation in the amounts of iron found in different optical glasses made in this country.

[1] Iron introduced into the batch during mixing is included here under that contributed by the raw materials; iron may also be introduced mechanically from the furnaces.

The amount contributed by the batch and also the amount absorbed from the pot are given.[1]

A considerable part of the iron introduced into the glass from the pot comes from the complete solution of a portion of the pot; that is, either the solution of a uniform layer from the inside surface, or the solution of pot "stones." Another part comes from selective action[2] of the batch or glass on the pot material; iron oxide, silica, and perhaps other oxides going into solution, while the remainder of the undissolved pot material crystallizes as sillimanite. The white line frequently seen between the glass and the darker portion of the pot material consists of a crystalline layer of sillimanite with interstitial spaces between the sillimanite fibers filled with glass. Such zones are usually less than a millimeter thick. Melts made in some pots showed also a zone of brown glass a few tenths of a millimeter thick in contact with the sillimanite layer; other similar pots of the same iron content did not show such a zone.

It must be emphasized that under some conditions a pot containing over 2 per cent iron oxide may actually contribute less iron to the glass than a pot containing less than 1 per cent. In other words, the physical condition of the pot plays an all-important role in resistance to pot corrosion. If a dense surface has been made on the interior of the pot and this has been thoroughly vitrified in the arching and burning processes the pot becomes exceedingly resistant and withstands the action of the batch;

[1] It should be noted here that the figures for iron introduced from the batch are higher than would result from materials now available. The experiments recorded here were all carried out before October, 1917, since which time considerable progress has been made in the development of better raw materials.

[2] Microscopical evidence on this selective action has been given by Bowen (*J. Am. Ceram. Soc.*, 1, 596 (1918)). The chemical evidence is simply that whereas the pot material may contain 10 or 15 times as much alumina as iron oxide, the glasses made in such pots show a much smaller ratio between these two oxides. Reference to the analyses made by Allen and Zies (*J. Am. Ceram. Soc.*, 1, 784 (1918)) of American and German optical glasses shows that the *maximum* ratio $Fe_2O_3 : Al_2O_3$ is 1 to 5.5 (with the exception of Jena O — 1209 the batch of which contains alumina). Our own analyses on other optical glasses show similar small ratios, as do also some unpublished determinations of our colleague, Dr. H. S. Washington.

under such conditions, the amount of iron absorbed from the pot may be reduced to 0.02 per cent or less in the glass.[1] On the other hand, pots containing smaller amounts of iron may introduce more iron into the glass because of poor condition of the interior—such as high porosity, cracks around grog particles, crazing, etc.—which encourages pot attack.

TABLE 1.

Iron Absorbed from Pots by Optical Glasses.

Glass type.	Approx. Fe_2O_3 content of pot.	Fe_2O_3 from batch.	Fe_2O_3 in glass.	Fe_2O_3 from pot.
CROWN:				
Borosilicate.....	2.2 %	0.030%	0.080%	0.050%
Borosilicate.....	?	0.022	0.035	0.013
Light.....	3.0	0.025	0.074	0.049
Light.....	2.5	0.025	0.038	0.013
Light.....	0.5	0.025	0.031	0.006
Light Barium.....	2.2	0.027	0.053	0.026
Light Barium.....	2.2	0.027	0.075	0.048
Light Barium.....	0.75	0.035	0.043	0.008
FLINT:				
Baryta.....	0.75	0.025	0.035	0.010
Dense.....	2.2	0.017	0.056	0.039
Dense.....	0.75	0.017	0.020	0.003
Light.....	2.2	0.020	0.053	0.033
Light.....	2.2	0.020	0.037	0.017
Very dense.....	2.2	0.022	0.062	0.040

The weight percentage of iron introduced into the glass from the pot varies considerably, other conditions being equal, with the size of the pot and the density of the glass; the surface exposed to the glass increases as the square of the diameter while the capacity goes up as the cube. The weight percentage of iron is highest in the lighter glasses such as the borosilicate and light crowns and lowest in the very dense flints. These relations are made clear in Fig. 1, which gives the calculated percentage of

[1] The pot shown on the left in Fig. 5 contributed only 0.017% Fe_2O_3 to the light flint made in it. Note the sharp line of demarcation between pot and glass. The pot carried 2.2% iron oxide.

Fe_2O_3 which might be introduced into glass from the pot for different types of glass in pots of the usual sizes and proportions. The figures given here are based on the solution of all the iron from a uniform layer 1 mm. thick over the entire inner surface of the pot, and upon an iron oxide content of the pot taken as 2 per cent. Actually it has been found that pot corrosion is generally greater around the knuckle of the pot and across the bottom than around the sides. With dissolution of a millimeter layer in the 36-inch pot ordinarily used and an assumed iron content of 2 per cent Fe_2O_3, the percentage of iron absorbed from the pot

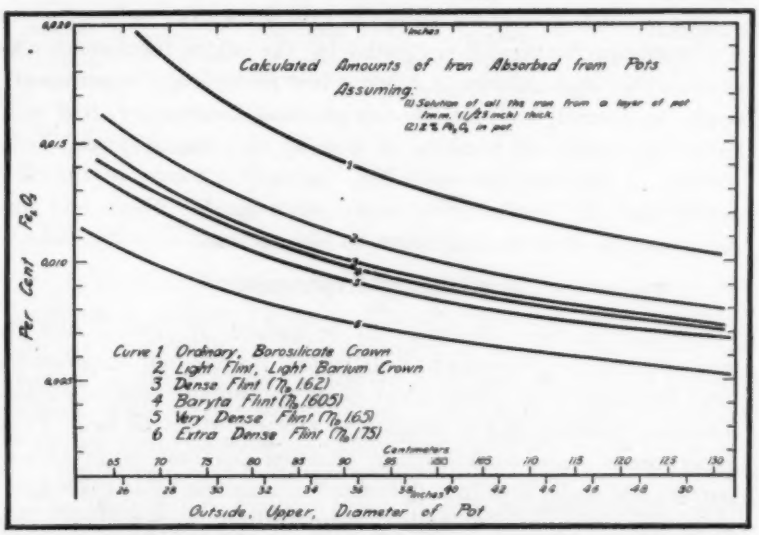
Calculated Amounts of Iron Absorbed from Pots
Assuming:
(1) Solution of all the iron from a layer of pot 1 mm. (1/25 inch) thick.
(2) 2 % Fe2O3 in pot.
Per Cent Fe2O3
0.020
0.015
0.010
0.005
Curve 1 Ordinary, Borosilicate Crown
2 Light Flint, Light Barium Crown
3 Dense Flint (nD 1.62)
4 Baryta Flint (nD 1.605)
5 Very Dense Flint (nD 1.65)
6 Extra Dense Flint (nD 1.75)
Centimeters
65 70 75 80 85 90 95 100 105 110 115 120 125 130
26 28 30 32 34 36 38 Inches 40 42 44 46 48 50
Outside, Upper, Diameter of Pot

FIG. 1.—Calculated percentages of Fe_2O_3 in various glasses, derived from various sizes of pots, on the assumption of complete solution of the iron in a layer 1 mm. thick, and a ferric oxide content in the pot shell of 2.0 per cent.

is 0.014 per cent for a borosilicate or light crown and 0.010 per cent for a flint of index 1.62. The surface of the clay stirring tube exposed to the melt has not been considered in these calculations, although this surface is about 10 per cent of the inner surface of the pot. Under actual conditions the amounts of iron oxide absorbed from the pot are frequently several times those given in Fig. 1. (See Table 1.)

Use of Chlorides, or Chlorine, to Volatilize Iron.—The use of various chlorides for volatilizing iron from enamels is familiar, and has been described by Bole and Howe,[1] who found that from 8 to 36 per cent of the total iron could be removed from batches by this method. A method for the purification of sand has been described,[2] which is based upon the ignition of the sand with 2.5 per cent sodium chloride. The use of chlorine for the volatilization of certain metals is of frequent occurrence in analytical chemistry and similar reactions are taken advantage of in the purification of various compounds. Chlorine at high temperature has recently been used by Phillips to purify zirconia from iron.[3]

The possibility of volatilizing the iron from a pot before the pot was used was naturally suggested by the above-mentioned uses of chlorides and chlorine. After a few preliminary experiments, made on a small scale at the Geophysical Laboratory, had indicated the successful removal of iron by this method, one of the writers, in conversation with Mr. George C. Stone, of the New Jersey Zinc Co., learned that many years ago Mr. Stone had similarly used a stream of chlorine to bleach small clay crucibles.

Preliminary Experiments.

Recognizing the advantage to be gained by diminishing the iron content of the available pots if only by one-half, experiments were first made on a laboratory scale to find the best conditions for using chlorine and to learn how efficient it might be. These experiments were conducted by heating portions of previously burned pot shell, held in a porcelain or alundum boat placed in a silica glass (or, at the higher temperatures, a porcelain) tube through which the chlorine stream was forced. The tube was heated in an electric furnace and the temperatures, taken by a platinum platinum-rhodium thermoelement, were read on a millivoltmeter. The first experiments showed that chlorine at 800° C[4] would remove some portion of the iron but that better results were obtained at higher temperatures. The effect of

[1] *Trans. Am. Ceram. Soc.*, 17, p. 125 (1915).

[2] J. G. A. Rhodin, Brit. Patent No. 8495 (1914).

[3] A. J. Phillips, *J. Am. Ceram. Soc.*, 1, 791 (1918).

[4] All temperatures in this paper are expressed in °C.

water vapor was later tested by passing the stream of chlorine through water before introducing it into the tube; the effect of hydrochloric acid gas was similarly tested; the results of these experiments will be given below.

Effect of Time and Temperature.—The details of several runs under different conditions are given in Table 2. From the results of these tests it is evident that the chlorine treatment should

TABLE 2.

Iron Removed from Pot Shell by Chlorine.
(Chlorine Flowing from Zero Time).

Minutes to reach maximum temperature.	Maximum temperature °C.	Minutes held at maximum temperature.	Iron content of pot shell % Fe_2O_3.		Percentage of total Fe_2O_3 removed by Cl.
			Initial.	Final.	
...	1100°	120[1]	2.19	1.03	53.0
155	1214	195	2.19	0.78	64.3
...	1225	105	2.19	1.20	45.2
185	1365	125	2.19	0.49	77.6

be conducted at the highest attainable temperature and should continue for at least 2 hours. The pot shell used here contained 2.19 per cent Fe_2O_3.[2] The residual iron, after a three-hour run at 1214°, is about 36 per cent of the total amount of Fe_2O_3 initially present, while after 2 hours at 1365° the residual iron is reduced to 22 per cent, showing conclusively that from $^2/_3$ to $^4/_5$ of the iron can be removed by this method.

The treated pot samples after these runs were nearly white in color with occasional yellowish spots scattered through the mass. The portions of pot were upwards of a centimeter in thickness and no difference in color was to be seen between the outer and interior portions showing that the gas had penetrated to the center. The appearance of the treated pot shell re-

[1] Approximate time.

[2] All results in this paper on iron contents of pots and glasses are calculated as per cent Fe_2O_3 without reference to the actual state of oxidation of the iron. Analyses by J. B. Ferguson, to be published later, show that glasses made in gas-fired furnaces may carry from 10 to about 30 per cent of the total iron in the ferrous state.

sembled very closely certain of the more refractory kaolin pots developed later. Some pieces of ordinary red brick were placed in the boat along with the pot shell in one run and after the treatment with chlorine they were beautifully white; the results of the chlorine treatment are evident from Fig. 2, which shows portions of treated and untreated brick. In another experiment an "Alundum" boat was used as the container for the pot shell; it also was bleached to a fine white, which did not show minute dark specks under the microscope as is frequently the case with alundum ware ignited in air.

Effect of Water Vapor.—Inasmuch as the products of combustion in a gas-fired furnace might interfere seriously with the volatilization of iron by this method it was desirable to investi-

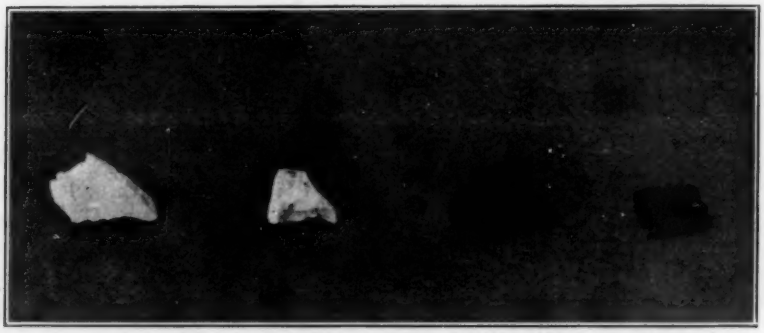

FIG. 2.—Photograph showing treated and untreated ordinary red building brick.

gate the possible effects of such gases upon the reaction. Of the ordinary products of combustion, moisture might conceivably be the most objectionable since it would decompose the ferric chloride vapor with the formation of Fe_2O_3 and HCl.

The effect of water vapor on the reaction was studied by making a run with chlorine, which, previous to its introduction into the heated tube, was passed through warm water. A check run was made with dry chlorine. The results are given in Table 3. It is seen that whereas dry chlorine takes out 61.8 per cent of the

TABLE 3.

Effect of Moisture and of HCl on the Volatilization of Iron by Chlorine.

Chlorine.	Time at maximum temperature.	Temperature °C.	Iron content of pot shell, per cent Fe_2O_3.		Per cent of total Fe_2O_3 removed.
			Initial.	Final.	
Dry	30 min.	1265	1.94	0.74	61.8
Bubbled through water at 69°	30 min.	1265	1.94	0.88	54.7
Bubbled through conc. HCl at 17°	30 min.	1265	1.94	0.84	56.7

iron; chlorine carrying moisture, under identical conditions of time and temperature, takes out 54.7 per cent. These two figures are not so far apart as had been expected.

Effect of Hydrochloric Acid Vapor.—Prof. H. H. Willard, of the University of Michigan, suggested the possibility that hydrochloric acid might assist in the reaction and accordingly a run was made in which the chlorine was bubbled through concentrated hydrochloric acid before being introduced into the heated tube. The effect was here almost identical with that obtained with moist chlorine. The percentage of iron removed with chlorine and hydrochloric acid vapor was 56.7 per cent as against 61.8 per cent for pure chlorine and 54.7 per cent for chlorine and water vapor. There is no advantage to be gained therefore under these conditions from the use of hydrochloric acid vapor[1] along with chlorine. The details of the run with hydrochloric acid and chlorine are given in Table 3.

Large Scale Experiments.

The laboratory experiments having thoroughly demonstrated that pot shell could be freed of the greater portion of its iron by a simple treatment with chlorine at temperatures easily secured in glass furnaces the experimentation was next transferred to the glass plant of the Bausch and Lomb Optical Co., which very kindly

[1] Dry hydrochloric acid gas may be beneficial, its use, however, would require additional equipment (for generation and drying) which would not be readily adaptable to furnace hall operations.

placed equipment at our disposal. It is also fitting at this ti me to express our thanks to Mr. Victor Martin, glassmaker in charge, for the kind assistance which he rendered us.

These large scale experiments were carried out in order to learn the optimum conditions for volatilizing iron from glass pots by means of chlorine, and after these conditions were established, to chlorinate pots and study the quality of glass melted in pots so treated. The several runs will be described separately and full details given.

Necessary Equipment.—The equipment required for factory experiments was a source of chlorine (a cylinder of liquefied gas), a wash bottle containing concentrated sulfuric acid[1] connected to the cylinder to show the rate of flow of the gas, and a rubber tube connecting the wash bottle to a 15 mm. fused silica glass tube about 200 cms. long which served for the introduction of the gas into the heated pot. Furnace equipment of different types was at hand and some of the pots were treated in a recuperative furnace used for experimental work, others in pot arches, and one in a regenerative furnace. Temperatures were read with an optical pyrometer which was checked against thermoelements.

Treating a "Thread" Pot.—The first experiment on treating a "thread" pot was carried out in the recuperative furnace. In this test the pot was turned bottom up and chlorine was introduced beneath the pot. This test was qualitative only and no accurate record was kept of the amount of gas used, or the time of exposure. The temperature was kept at approximately 1200° and after treatment the pot was allowed to cool with the furnace. On examination next day it was found that the pot was noticeably bleached around the upper zones which, because of the relative position in the furnace, had been subjected to the most vigorous action of the gas. A deposit of beautiful hematite crystals was found around the edge of the pot where the ferric chloride vapors had come into contact with the furnace gases.

[1] Other liquids might have been used here but in choosing a liquid to show speed of gas it should be remembered that water vapor is to be avoided and sulfuric acid not only contributes no water but acts as a dehydrating agent.

The second experiment was also made with a "thread" pot covered somewhat as in Fig. 3A; the gas being introduced into the pot as shown in the figure. Chlorine was passed for two hours and again the temperature was around 1200°. The pot was cooled in the furnace as before and when cold was removed from the furnace and examined. In this case the pot was thoroughly bleached, as was also the fire clay slab used for a cover. Fig. 4 shows a photograph[1] of a section of the broken pot; the bleached zone may be readily seen. Deposits of hematite crystals were present around the upper portion of the pot and on the under side

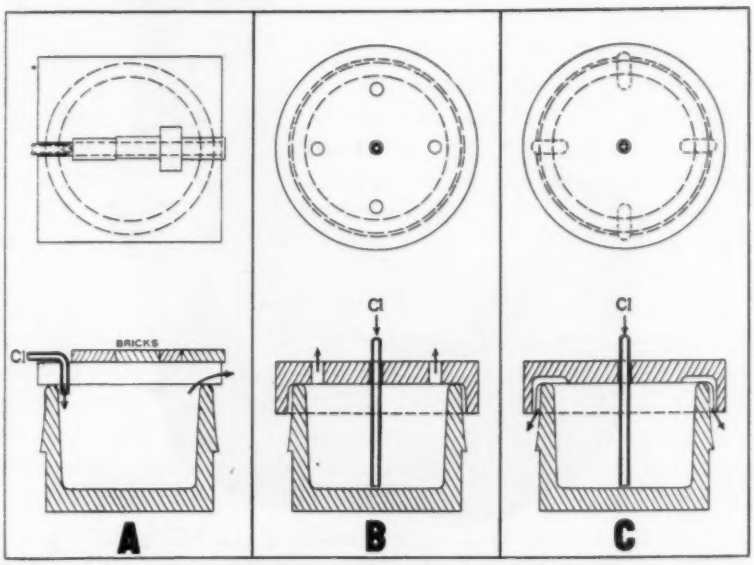

FIG. 3.—Plans and vertical cross-sections of pots, showing various methods of introducing chlorine.

of the cover where the effluent gases were met by the products of combustion in the furnace.

This test showed the practicability of the method as applied to large scale work.

Test on a Large Pot. Melt Number 573.—The next test was made on a type of pot used regularly at that time for the melting

[1] By Dr. R. H. Lombard, of the Geophysical Laboratory.

of optical glass and was carried out in one of the regenerative furnaces. The pot was covered in the manner shown in Fig. 3A and the chlorine stream was introduced through a silica glass tube bent as shown in the sketch. The pot was subjected to the action of chlorine for 2 hours at a temperature of 1225° when the fire-clay cover was removed and the furnace taken up to the filling temperature. When the cover was removed a cloud, which

FIG. 4.—Photograph of a section of a chlorinated pot showing depth of penetration.

probably consisted of hematite crystals, was seen to form within and about the pot and hence the results obtained later on the amount of iron extracted from the pot by the glass melt were rendered somewhat uncertain. The cover showed a beautiful deposit of hematite crystals where the chlorine and ferric chloride made their exit from the pot. Some of the crystals were 5 mm. on a side; they will be described in detail elsewhere.

A melting of dense flint was carried out in this pot immediately after the treatment with chlorine. Two other melts (Numbers

571 and 572) with similar (but untreated) pots and portions of the same batch were made at the same time in the other furnaces in order to have a check. The iron introduced into the glass from the batch was in all three cases 0.017 per cent Fe_2O_3. The glass from Melt 571 was later found to contain 0.056 per cent Fe_2O_3 and Melt 572 analyzed 0.055 per cent, showing that 0.038 per cent had been absorbed from the untreated pots. The iron content of Melt 573 was found to be 0.048 per cent Fe_2O_3, which means that the amount absorbed from the treated pot was 0.031 per cent. This difference, although less than had been expected, was nevertheless encouraging.

Experiment with a Large Pot in an Arch.—The experiment just described seemed to indicate that the chlorination had not been thoroughly effective for perhaps two reasons: first that since the chlorine had been introduced into the pot through a tube that did not extend much below the cover, the upper portions only of the pot had been freed from iron; and second that the precipitation of ferric oxide above mentioned had introduced some portion of these crystals into the pot where they were later dissolved by the glass. The next steps seemed, therefore, to be (1) to introduce the chlorine stream in such a manner that it first impinged upon the bottom of the pot, in order to remove iron where the greatest corrosion takes place during melting; and (2) to control the direction of the effluent gas so that precipitation of the ferric oxide formed by interaction of the ferric chloride with the furnace gases would not occur within the pot. Inasmuch as these conditions could not be readily met in the furnaces the next experiments were carried out in the arches used for drying and preheating the pots. The construction of these arches permitted greater latitude for manipulation than the furnaces, but on the other hand the maximum temperature attainable was considerably less.

The method of treating the next pot is indicated in Fig. 3B, which shows the entrance tube for the chlorine, reaching nearly to the bottom of the pot, and the method of covering. The pot used for this test was one which had been discarded because of cracks; it was of the usual 27-inch type. After the arch had been

heated to 1075° the pot was chlorinated for 2 hours at this temperature; the amount of chlorine used was 2.5 pounds. The pot was later taken out and examined. A deposit of hematite crystals had formed on the outer surface of the pot. The bleached portion of the pot was clearly evident in the cross section, and it was found that the bottom of the pot was bleached to a depth of about 15 mm., while the whitened zone became thinner towards the top. The result of this test was very satisfactory, therefore, in showing the best conditions for bleaching.

When the pot was taken into the sunlight, for purposes of photographing, the interior surface was seen to be covered with small glistening points. Examination under a hand lens showed them to be minute diamond-shaped crystals which were shown later by chemical and microscopical tests to be titanium dioxide. These crystals were studied in detail by Dr. H. E. Merwin and his results will be published in another place.

Chlorination in Arch. Melt Number 599.—The last experiment having demonstrated that the pot could be thoroughly bleached in those portions deemed most necessary, another pot was treated in an arch for 3 hours at 1007°, using the method of covering and of introducing the chlorine shown in Fig. 3B. Three pounds of chlorine were passed into the pot. Owing to accidental circumstances the pot stood in the arch four hours after it had been treated before being transferred to the furnace. Flames played over the pot during its treatment with chlorine and also during the period immediately before its transfer to the furnace.

A light flint batch was now melted in this pot, following the usual procedure. The batch contained iron equivalent to 0.02 per cent Fe_2O_3 in the glass. A proof taken during the fining process (while seeds were still present) showed 0.037 and the final glass[1] gave 0.048 per cent Fe_2O_3. Glass from the same type batch made in similar but untreated pots showed iron values from 0.053 to 0.058 per cent Fe_2O_3.

The bleached zone in contact with the glass is shown on the

[1] The absorption of light of Melt Number 599 was later found to be 1.4 per cent per cm. as determined by the Scientific Bureau of the Bausch and Lomb Optical Company.

right in Fig. 5; the brown glass layer, frequently observed in these pots, was absent.

The disappointing result obtained with this run was initially attributed to several factors. In the first place, the temperature

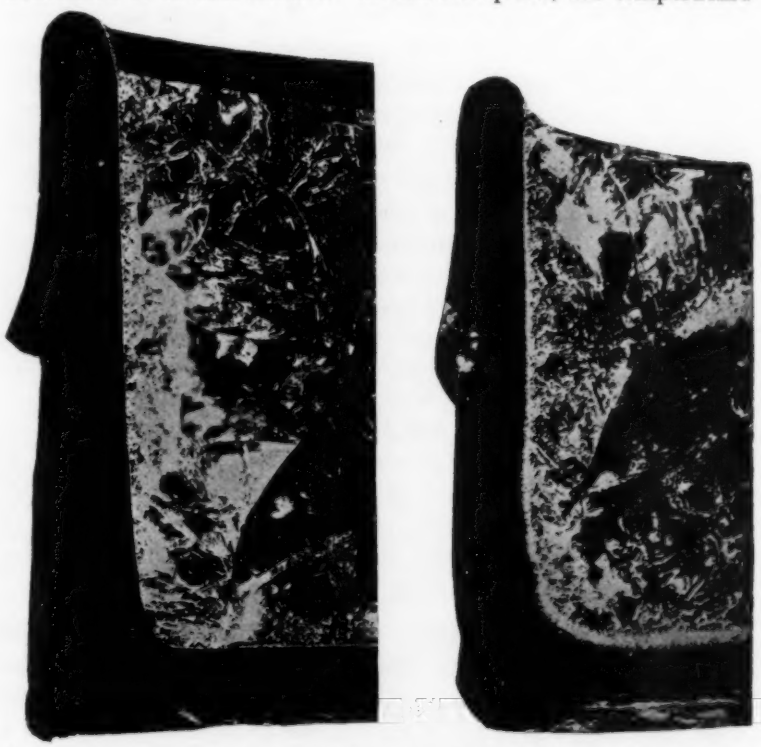

FIG. 5.—Photographs of cross-sections of pots containing melts, showing bleached (chlorinated) zone in the treated pot on the right. (Photo on left by Bausch & Lomb Optical Company; photo on right by Dr. F. E. Wright, Geophysical Laboratory.)

during chlorination was only 1000° and the preliminary experiments showed conclusively that considerably more iron would have been volatilized at a higher temperature. Much more serious appeared to be the fact that the pot, after the flow of gas had ceased, had stood filled with chlorine in the arch at the temperature of chlorination for four hours. During this period the

pot was surrounded with a reducing atmosphere which, penetrating to the interior of the pot, would tend to form ferrous chloride. The concentration of ferric chloride vapor would also increase, inasmuch as reaction between iron oxide and chlorine would continue at this temperature. When the cover of the pot was later removed—previous to the transfer to the furnace—a precipitation of ferric oxide would occur inside the pot and eventually this oxide would dissolve in the glass. The iron oxide precipitated under these conditions would probably not be wholly ferric but, under the reducing conditions obtaining here, it would contain ferrous oxide in solid solution.[1] The latter oxide would tend to produce a darker color in the glass.

Another possible cause suggested to us for the high amount of iron in the final glass was the presence in the pores of the pot of ferric chloride which was later absorbed by the glass. The following calculation shows, however, that such a condition would not increase the amount of iron in the glass to an unusual extent. The volume of pot walls and bottom may be taken roughly as 100 liters for the smaller size of pot used. Let us assume, in order to have the maximum value, that the pore space is 25 per cent. At 1000° the weight of ferric chloride vapor, assumed to be Fe_2Cl_6, occupying 25 liters, is about 90 grams, which is equivalent to 0.007 per cent Fe_2O_3 in the melt of average weight.

Large Pot Treated in Arch. Melt Number 610[2].—In order to avoid the possible difficulty caused by allowing an atmosphere of chlorine and ferric chloride to remain in the pot after treating with chlorine the next pot so treated was finally swept out with a stream of nitrogen from a cylinder of compressed gas. Fig. 3C shows the method of introducing the chlorine and also the method of covering the pot. In order to avoid the possible precipitation of hematite crystals where gases passed through the openings in the cover used previously (Fig. 3B), channels were cut in the under side of the cover, as shown in the figure, so that the precipitation would take place outside the pot. This pot was chlori-

[1] R. B. Sosman and J. C. Hostetter, "Solid Solution in the System Fe_2O_3–Fe_3O_4," *J. Am. Chem. Soc.*, **38**, 807–833, 1916.

[2] This test was carried out by our colleague, Dr. N. L. Bowen, to whom our thanks are due.

nated in an arch at 1075° for 5 hours; 7.5 pounds of chlorine were used at a uniform rate of flow. A light flint batch was afterwards melted in this pot but the result was not satisfactory. The melt contained many pot stones and there were other evidences of pot attack.

When the pot was later broken down the portions of the pot in contact with the glass were found to be thoroughly honeycombed, shelling off in layers and showing penetration by the glass to a depth of several millimeters.

Analysis of the glass from this melt showed the presence of 0.067 per cent Fe_2O_3, while the companion melt made as a check showed only 0.053 per cent. The inner layers of the pot were thoroughly bleached and this fact considered in connection with the high amount of iron found in the glass indicated that much more pot attack had taken place than normally. This was later confirmed by a determination of alumina in the glass from the treated pot and also from the check pot; the former carried 0.143 per cent Al_2O_3, while the latter contained only 0.067 per cent. This shows that at least 2 times as much pot dissolved from the shell of the treated pot—actually the amount of pot disintegrated was probably many times that from the untreated pot, because, as stated previously, the dissolving action of the melt is selective to a certain extent and relatively more iron than alumina is taken into the glass, a large proportion of the alumina persisting as pot stones made up of sillimanite crystals.

Cost of Chlorinating Pots.

At 20 cents a pound for chlorine the cost of volatilizing iron from a pot is of small moment. The maximum used in any chlorination (Melt No. 610) was 7.5 pounds at a cost of $1.50. The other necessary apparatus is cheap and the technic not difficult to carry out. If the process were adopted a permanent set-up could be made for each arch, or furnace, so that the chlorine could be readily connected and the entrance tube lowered into the pot. Hooded pots would present even less difficulty, as far as covering is concerned, than open pots.

Concluding Remarks.

The experiments recorded above point conclusively to the fact that iron can be volatilized from glass pots under factory conditions and that there is nothing difficult or impracticable in carrying out the process. The experiments also showed that the amount of iron in the pot could be readily reduced by half. The final tests showed less iron in the glass made in treated pots than in the glass made in untreated pots in all cases but one (the last test described above). The evidence all points toward the conclusion that the removal of iron from these pots—pots which depended in part upon iron as a bonding element—made them more porous and hence more readily attacked by the melted batch. Possibly these same pots would have been sufficiently dense even after chlorine treatment if a different burning schedule had been in operation at the time the experiments were made. The usual practice at that time was to set the pot in the furnace at 4 P. M. and to heat to 1400° by 7 or 8 o'clock. Later, a burning period of several hours at a higher temperature was added to the schedule, but no other experiments on volatilizing iron were made.

The discontinuance of these experiments, at a time when the many difficulties inherent in the early development of a new process bid fair to be completely solved, is regrettable, but was necessitated by the imperative demand for immediate maximum production of glass, rather than for the production of a glass of maximum transmission at a temporary sacrifice of capacity. Our experiences recorded here indicate that the process would function successfully with a pot in which the iron, though present, is not one of the bonding elements. Our later experiences in the arching and burning of pots make us confident that with proper heat treatment the disintegrating effect of the chlorine upon the pots in which iron is an essential bonding element could easily be overcome. In addition the application of this process to pot material, such as grog, would appear feasible, and this application alone would reduce the amount of iron in the final pot by one-third to one-half.

GEOPHYSICAL LABORATORY,
CARNEGIE INSTITUTION OF WASHINGTON,
WASHINGTON D. C., April, 1919.

DISCUSSION OF AN ARTICLE BY G. W. MOREY ENTITLED "AN IMPROVED METHOD OF OPTICAL GLASS MANUFACTURE" IN THE FEBRUARY NUMBER OF THE JOURNAL OF THE AMERICAN CERAMIC SOCIETY.

By Clarence N. Fenner.

The article by Mr. Morey on "An Improved Method of Optical Glass Manufacture" in the February number of the JOURNAL describes an interesting and valuable advance in the manufacture of optical glass. I think, however, that a word of caution may well be kept in mind. The inference from Mr. Morey's paper would seem to be that the process is applicable without restriction. That inference might at times lead one into difficulties. A circumstance which would be likely to render a longer period of fining advantageous or even essential would be the presence in the batch materials of those impurities, such as sulfates and chlorides, which tend to cause some glasses to become milky or opalescent. The presence of more than 0.30 per cent SO_3 or more than 2.0 per cent Cl in the potash used is likely to be dangerous (with some flints especially) and to make it desirable to hold the glass at an elevated temperature for several hours after melting is complete. In the early work of the men of the Geophysical Laboratory at the Bausch and Lomb works, milky glass was one of the chief evils with which they had to contend, and the amount of work which was devoted to finding out the cause of the phenomenon and learning what procedure was necessary in order to avoid it has left a strong impression with those who were engaged upon the optical glass work at that time, so that the possibility of the recurrence of such conditions is likely to be present in their minds. Later it was found possible to cut off the source of the trouble by requiring that the potash furnished by the manufacturers should meet definite specifications, based on the above-mentioned experience. It was only then, I think, when assurance could be had of a supply of pure raw materials, that shortening the fining process became feasible. It would be well for a manufacturer of optical glass, when considering the use of a twenty-four-hour schedule, to bear such limitations in mind.

Geophysical Laboratory,
Carnegie Institution of Washington,
Washington, D. C., April, 1919.

NOTES ON FIRE CLAYS OF THE NORTHERN APPALACHIAN COAL BASIN.

By Ellis Lovejoy, E.M., Columbus, Ohio.

The chief flint fire-clays in the northern Appalachian coal basin are:

(1) The Sciotoville in Kentucky. This is the Sharon horizon in Pennsylvania but in Ohio is known as the Sciotoville or No. 1.

(2) The Mercer in Pennsylvania and Maryland.

(3) The Lower Kittanning in Ohio (No. 5), Pennsylvania and Maryland.

(4) The Upper Freeport in Ohio (No. 7), Pennsylvania and Maryland.

The Brookville (No. 4 in Ohio) is reported to have excellent deposits of flint clay in Pennsylvania, and several other horizons are noted as bearing flint clay but none furnish any high grade material, or at least very little for the fire brick industries. The Brookville horizon has no flint clay in Ohio but it is an excellent plastic clay. In several localities in southern Ohio it has a peculiarity which has assisted the writer in working out some geological sections. In the localities mentioned, the outcrop does not tail down in the usual clay blossom but instead crumbles out in fine angular fragments and at a distance the waste resembles yellow sand, or more nearly sawdust. Close examination reveals the fact that each tiny fragment is conchoidal and in every visual respect a flint clay.

The Sciotoville clay is largely mined in Olive Hill, Kentucky and vicinity, and no where else so far as I know. It is a light-colored clay and in texture and fracture a typical No. 1 quality flint clay. This clay in relatively large areas has disseminated through it minute concretions of sulphide of iron, ranging in size from that of a pin head to microscopic grains. In some blocks the grains of pyrite are so small as to be practically invisible and their presence in detrimental quantity can only be determined by burning. This flint clay is found in pockets as far north as Logan, Ohio,

but in such pockets it is in the form of a boulder clay. The normal deposit has plastic clay immediately under the coal, underlaid by flint, and under the flint comes the semi-hard clay, but there is wide variation in the deposit. Not only is there variation in the thickness of each bed and in the quality of each clay but any one or two of the three may be entirely lacking.

The Mercer flint clay, sometimes designated as the Conglomerate clay in Pennsylvania because of its position relative to the Homewood and Connoquenessing sandstones is the most widely used and in general the best in the Appalachian basin. It is not found in Ohio, nor Kentucky, at least not in workable thickness, but it is extensively mined in central Pennsylvania and in the vicinity of Cumberland, Maryland. The bed cannot be said to have any typical structure. In the southern field it occurs in two benches with a division band of a few inches of sandstone or sandy clay. The benches vary up to fifteen or more feet in thickness with the flint clay in pockets embedded in the plastic. In central Pennsylvania the bed is normally thinner and is worked as low as three feet, including the plastic and "Burley" clay. The flint clay is more often found in the upper part of the bed, but so erratic is it in its position in the bed, that one is not justified in assigning it a normal position.

The Pennsylvania Geological Survey[1] sums it up as follows: "In the thick bed of plastic clay the flint clay may occur in any position in the same mine, sometimes at the bottom, sometimes at the top, or in any intermediate position, sometimes running out altogether, sometimes forming the whole bed, though as previously stated, there is almost invariably at least an inch or two of soft clay between the flint clay and an overlying coal bed, if the clay, as generally is the case, be overlain by coal."

I do not know the variations of the clay, but in so far as my observation goes, it frequently is widely different from a characteristic flint clay. It is harder than the other flint clays and some of it weathers down very slowly. In fact, some of it cannot be disintegrated by weathering within a practical period of time. Some portions of the bed lack the characteristic conchoidal frac-

[1] Topographic and Geologic Survey of Pennsylvania. p. 317, 1906–08.

ture and have not the dense smooth surfaces. Instead it breaks out in irregular shale like blocks with a granular surface. One familiar with flint clays would mistakenly pronounce this rough, apparently coarse grained Mercer flint to be of doubtful quality because of its appearance. This granular clay has a nodular structure to which the grainy appearance is due, and it is claimed that this rough clay is quite as refractory as the characteristically flinty clay from the same bed.

The Mercer flint clay is generally dark gray in color, which serves to distinguish it from the other flint clay horizons but the color is not to be taken as conclusive.

The nodular structure is peculiar and interesting. The nodules are not unlike bauxite concretions or "crystals" and I sometimes wonder if they are not genetically related.

In the Cretaceous kaolin deposits along the Fall line in Georgia, and similarly in Minnesota, are found extensive development of nodules or bauxite "crystals" and the only apparent difference between the true bauxite and the bauxite "blossoms," as the bauxitic kaolin is locally called, is that the latter is chalk white, while the true bauxite is gray in color. I have had analyses of the "blossom" nodules but they show kaolin only.

These nodular kaolins under cover are quite soft but have very little plasticity. They are readily cut out with an ax or saw and shaped up for building purposes and harden to a durable stone upon sufficient exposure.

The thought comes to me that the nodular kaolins, closely associated as they are in Georgia with bauxite deposits, have been subjected to the same conditions which developed the bauxite, except that the dissociation of the kaolin is only partial; that the silica set free by the dissociation has not been removed and to this soluble silica is due the subsequent hardening. An analysis naturally would show a kaolin ratio although the nodule might not be kaolin. I was told that the nodules in the flint clay in the Mercer horizon were higher in alumina than the normal clay which has practically a kaolin ratio, but an analysis which I made did not confirm this.

The Mercer clay beds which are worked in the Clearfield, Pa. district carry very pure flint clays and in my opinion rank first in

quality compared with any flint clays in the Appalachian basin. Farther east in the Lock Haven district the Mercer clay runs higher in iron but not to such an extent as to materially affect the quality of the product.

The Lower Kittanning flint clay is worked in Jackson county, Ohio, especially in the vicinity of Oak Hill, and extends north into Vinton county. Occasionally traces of it are found in central-eastern Ohio but it is not workable and is lost sight of in the vast quantity of excellent plastic clay at this horizon. Its best development in Ohio is in Tuscarawas county, northern Ohio, north of Canal Dover, around Mineral Point and particularly near Strasburg.

It is found in central Pennsylvania where it is largely used in the manufacture of fire bricks, but it is not the equal of the Lower Mercer clay and it is only used where the latter is not available. I have also found it in good thickness in Georges Creek, near Cumberland, Maryland.

It is not so hard and flinty as the Mercer or the Sciotoville and in color is neither a cream-buff nor a gray but instead an intermediate drab. Where mined near the outcrop it has a brownish color, probably due to extraneous iron, and in the mass a mottled-greenish cast.

The mottled color of this flint clay is evident in a number of localities. In Georges Creek above mentioned the flint clay is made up of a mixture of light and dark gray, or more nearly a matrix of dark gray clay in which are embedded angular masses of the light clay—all flint clay with no physical line of separation to account for or distinguish the two clays. It is my opinion that there is a difference in the two and that as the outcrop is approached the matrix is disintegrated to a soft clay, perhaps not plastic and in this event it would be the so-called semi-hard clay, in which remain the boulders of undisintegrated light-colored clay. The flint clay from this horizon in southern Ohio also has this variegated color, and thus we have this characteristic in two widely separated districts in Maryland and Ohio. I do not know whether the color peculiarity prevails in the Pennsylvania districts or not, but I am told that it does in some localities.

A question might be raised whether the Oak Hill clay should be identified as the Lower Kittanning, since it is found on top of the coal of that horizon, while the plastic clay for the manufacture of bricks in that locality comes from under the coal. The old theory that the coal grew on the clay bed precludes the possibility of the clay being above the coal. There are a number of sections which are pinched, in which one clay with its cover of coal rests immediately upon a lower coal with its underlying clay, and if the intermediate clay is thick we may identify the two coals as separate horizons, or if the intermediate clay is thin we may count the two coal seams as a split seam in one horizon, particularly if the horizons above and below are in their proper place. Where two horizons are thus near together the removal of the upper coal would leave one coal with a clay above it and below it.

Near Hamden, Ohio, there are twenty-five feet of plastic and sandy clay at the Lower Kittanning horizon. Upon this rests a foot or more of coal, then a foot to a foot and one-half of flint clay, similar to the Oak Hill clay, and this is covered by a foot of coal. The Middle Kittanning and Upper Freeport coals are in their proper places higher in the hill. Here then we have the Lower Kittanning coal bed in about four feet thickness with a flint clay seam between the upper and lower benches of coal. There is nothing unusual in this except that the clay seam is a flint clay. We have, therefore, the Lower Kittanning flint clay above the coal at Oak Hill, within the coal at Hamden, and under the coal in northern Ohio, Pennsylvania and Maryland, in so far as I have seen it.

Wilber Stout's theory[1] that the clay beds accompanying the coals are hydrated coal ash, whatever objection may be raised to it, will explain many perplexing problems—one of which is the question of clay seams in coal beds.

The Middle Kittanning, Pittsburg hand Pomeroy coals in Ohio, each of which in its full thickness includes a clay seam from six to twelve inches thick, are not exceptional in this respect. They also include slate seams. The latter we can explain by floods, however brought about, covering the peat bogs with mud

[1] *Tran. Am. Ceram. Soc.*, **17**, p. 557 (1915).

which subsequently hardened into stratified shale which in coal beds we call slate. By analogy the clay seam is a similar flood deposit but not so stratified and quite different chemically. The stratification is of no consequence because a series of floods would give the stratified shale while a single deeper flood would give the clay deposit, but originally the same material as the shale.

We explain that the coal bed took root in the clay seam and purified it, particularly in the removal of iron since the shale is red burning and the clay buff.

We cannot understand why the coal should have grown on one mud deposit and not in another and why a thin streak of coal between clay and shale, as often happens, should have purified the seam of clay where a heavy bed of top coal had no effect on the shale.

Stout's theory of oxidation and hydration accounts for the clay seams without disturbing the evident cause of the shale seams, and the clay beds may be under, within, or on top of the coal or without accompanying coal, depending upon the vagaries of the oxidation.

It is not my purpose to discuss Stout's theory, and I only mention it because it fits in with my contention that the Oak Hill flint clay on top of the Lower Kittanning coal is the Lower Kittanning clay, and it also serves, though not essential, in a question which I will bring up later.

The Upper Freeport flint clay is characterized by iron carbonate concretions en masse. Thus I have seen it in Bolivar, Pa., Georges Creek, Md., in less degree in southeastern Ohio, and the Pennsylvania Geological Survey[1] mentions the presence of "ore balls" throughout a relatively large area in that State. These concretions vary in size from that of a marble to that of a barrel and in some cuts, where weathering has exposed them by crumbling away the clay, they are the predominating mass of the bed.

This flint clay is found in southern Ohio where the silica content is usually higher than in the other flint clays. In a number of localities where I have examined it, good clay is found in the outcrop but under cover it quickly merges into a sandy material,

[1] *Loc. cit.*, p. 320.

oftentimes scarcely better than a coarse sandstone. It seems likely that these outcrops are merely remnant boundaries of pockets of clay; that the mass of the clay has been removed by erosion up to the more resisting sandy material. In several localities, notably Moxahaha and the Carbon Hill, Ohio, the remnant is of workable area.

In recapitulation, in so far as my observation goes, the general

FIG. 1.—Specimens of Upper Freeport flint clay from the same mine.

No. 1. Blood-red flint clay.
No. 2. Ditto, burned to a buff color.
No. 3. Gray flint clay flamed with streaks of red, like "Aurora Borealis."
No. 4. Gray flint clay, veined.

characteristics of the four clays are as follows:

Sciotoville clay has a light color, close structure, conchoidal fracture and contains minute disseminated pyrite concretions.

Mercer clay is dark gray in color, exceptionally hard, weathers slowly, its more or less nodular structure giving it a granular appearance and this granular clay has not conchoidal fracture. The dense smooth portion of the clay is characteristically conchoidal.

Lower Kittanning clay is usually light, but frequently decidedly mottled in color; not so hard as either of the above nor so sharply conchoidal in its fracture; averages higher in silica,

which is frequently in the form of quartz grains embedded in the clay.

The Upper Freeport clay is light in color; contains accompanying iron carbonate concretions and frequently merges into very coarse sandy clay.

Physical Differences in Fire Clays.

It seems to me that Stout's theory above mentioned gives us a better starting point in an effort to explain the physical differences of plastic, "semi-hard," and flint clays.

Kaolins developed *in situ* are as a rule only slightly plastic, while alluvial kaolins and clays possess, in variable degree, greater plasticity and I think it cannot be controverted that the primary plasticity which these alluvia possess is due to the attrition which they receive during transportation. I use the word "primary" to designate the plasticity which a clay has when it is deposited and to distinguish it from any plastic development subsequent to such deposition. I am of the opinion that clays undergo changes in their plasticity after deposition. We know, of course, that plasticity is destroyed by heat and likely in some measure by pressure but, as I believe, we do not know that plasticity is increased following deposition.

All the shales of the coal measures are more or less plastic and while there are marked differences in the degree of plasticity, which cannot be fully accounted for by mineralogical differences, yet we get no such plasticity differences in the shales as we find in the fire clays, especially considering the close relation of the flint, "semi-hard," and plastic clays in each bed. The clays are practically chemically alike and it is inconceivable that the marked physical difference so sharply defined can be attributed to a primary cause; in other words, that these clays, if alluvial, came to their final resting place with the differences they now show.

It may be true, in fact it likely is true, that the pressure which has hardened the clays and shales, together with some heat development, has reduced the primary plasticity but we cannot ascribe to these causes the marked differences in contiguous flint and plastic clays.

Bacterial action has been frequently mentioned as effective in

developing colloidal conditions to which plasticity is ascribed and the presence of algae in clay has been given in explanation of the deep red color which some clays have. I have seen red fire clays which burned buff and the red color of the clay could not be due to iron, but instead was said to be in consequence of the presence of red algae. Notable examples of such red fire clay are found in the Upper Freeport horizon in southeastern Ohio. Numerous outcrops with a flaming red color are seen, and we also get pockets of red clay in the solid mass under cover, a thousand feet from the outcrop. All this red clay burns to a buff color and is quite refractory. Another peculiarity of flint clay in this locality is a marked venation, often broken and distorted, which seems to me not to be due to stratification.

The deep-red clays of Nova Scotia burn red but the burned color is not as deep a red as that of the raw clay, and this is also true of a deposit in Delaware which the writer developed several years ago. Whether the red color of these raw clays is in part due to algae, or not, I do not know, but in both localities the red clay is accompanied by muck clays favorable to the growth of bacteria or algae. In both instances these clays are extremely plastic. In boring into them with an auger, and in drawing the auger, the spiral of clay will frequently slip from the auger and the latter comes to the surface swept clean. When the auger holds its load of clay the latter streams out from the point of the auger in an attenuated string and may be loosened and slipped from the auger in a full length curl.

Some of the very red clays in the south burn a pale red, less deep in color than the raw clay. I am aware that these color differences in the raw and burned clay may be accounted for by iron solutions and agglomerations, except perhaps in the case of the fire clay mentioned, but the presence of algae offers a simple explanation.

In several instances I have found intimate irregular, I may say angular, mixtures of red and gray, or greenish gray clays. A notable example is in a clay mine about two miles north of Charleston, W. Va. The clay is fifteen to twenty feet thick and throughout it is an intimate mixture of red and greenish gray clay without any physical lines of demarcation between the two colors. In

the bottom of the bed is a fragmentary streak of flint clay. It was being mined under a solid roof without a trace of leakage and the mine was dusty throughout. It is inconceivable that the color difference extending to all parts of the bed without preponderance of either color in any part could be due to the oxidation of iron in the red patches. I am of the opinion that the color phenomenon of this bed is due to algae or other vegetable growth. Another instance is in southern Ohio where the peculiar appearance led the owner to some development work in search of precious metals.

If bacteria will develop in clay mixtures in storage cellars and increase the plasticity, as some potters claim, similar development will occur in clay bogs, and such bacteria, algae and other low forms of plant growths undoubtedly do occur in bogs whatever their effect on the clay mass. If a short period in a clay-storage cellar will materially increase the plasticity of a clay, we may expect a pronounced effect in the prolonged period of a geological accumulation.

Stout's theory of fire clays gives us a bog material primarily possessing little or no plasticity. It also explains the chemical differences of fire clay and shales, notwithstanding their juxtaposition. In these two factors it is favorable to this discussion, but not necessarily essential. We may assume that the fire clays and shales are transported materials; that the chemical differences are in consequence of the sources from which the materials were derived; that they possessed some primary plasticity, variable because of the variable material and the conditions of transportation; and finally that this plasticity was partially or completely destroyed by subsequent pressure with some heat development, or in other words, that the plasticity is destroyed to the extent that the material possessing little primary plasticity became non-plastic, while the more plastic material simply became less plastic.

With such a material in a bog teeming with protoplasmic growth, it is readily conceivable that there would be an extensive development of colloids, from the low forms of plant and animal life perhaps, and a colloidal condition in the clay material in consequence of the attrition of the grains through protoplasmic pressure and movement continued during a long period.

Protoplasmic growth is very sensitive to conditions and in the great area of plastic fire clays it is also conceivable that some small portion would be too pure to supply essential protoplasmic food, or that, because of acid, alkaline, or other chemical conditions, the protoplasmic growth would be retarded or prevented; that bordering this material there should be clays only feebly responsive to such cellular growth; and finally that, outside the influence of these unfavorable plots, the growth could be normal or excessive as conditions would permit.

It is not necessary to assume that there were no spores in any portion of the bed, but instead that some portions of the bed were unfavorable to their development and in consequence the colonization was slight. We would have, therefore, beds of light-burning clays with little initial plasticity. In some small portions there was no colloidal development and consequently no increase in plasticity; in other small portions colloidal development slightly increased the plasticity; in the great mass, however, over wide areas, the conditions would be favorable for protoplasmic growth with consequent colloids and resulting plasticity.

By subsequent pressure the bed is hardened into a rock and likely some measure of its plasticity destroyed, and this rock we are now mining and by grinding and pugging developing its latent plasticity—none in the flint, slight in the "semi-hard," and variably excellent in the plastic.

S. L. Galpin[1] presents a theory in explanation of the physical differences in fire clays diametrically the reverse of the writer's suggestion in the above notes. Galpin accepts the theory of sedimentary deposits and their purification by plant life, with the expressed opinion that these sediments were "largely colloidal." He starts with an exceedingly plastic mass whereas the writer's contention is that the mass is likely low in plasticity.

From this plastic material Galpin explains, that the flint clays are developed by setting and recrystallization of the colloidal sediments; that the "semi-flints" are derived from the flint clay through metamorphism by pressure and heat resulting in mineralogical changes; that the plastic clay associated with flint clay is

[1] S. L. Galpin, *Trans. Am. Ceram. Soc.*, **14**, 336–345 (1912).

weathered "semi-flint" clay and it is structurally and mineralogically different from other plastic fire clays which have never been "set."

COMMUNICATED DISCUSSIONS.

R. R. HICE: A review of the fire clays of the northern Appalachian coal basin is certainly most timely, and especially so in view of the enormous demands which have been made upon them, and the constant call for better and ever better fire-resisting materials.

In Pennsylvania, the most of the flint clays are produced from the Upper Mercer horizon. I think the variations in the Mercer clays have been well brought out by Mr. Lovejoy, and while it must be said some of these clays do not have the usual flint clay appearance, yet that fact does not take away their value as refractory materials, but is simply another complication to be taken into consideration in any satisfactory theory of the origin of flint clays.

I do not think the Lower Kittanning clay is worked in Central Pennsylvania to the extent indicated by Mr. Lovejoy. In Clearfield and Clinton Counties, for example, it is the Middle and not the Lower Kittanning clay which is used. In the clays underlying the three Kittanning coals, the occurrence of flint clay in the Lower Kittanning horizon is quite unusual. The promising flint clay horizons of the Allegheny series are the Middle Kittanning and the Bolivar. The Lower Kittanning is quite often used for bonding material, often being shipped quite a distance for that purpose. At one locality there is considerable flint clay in evidence but it is scattered throughout the bed in small nodules, seldom as much as one-half inch in diameter.

It is an obvious error to correlate the flint clay of Bolivar with the Upper Freeport horizon. The two beds are distinct and separate, not only at the type locality, but at other points as well. Forty years ago Stevenson, in describing the section at Bolivar, distinctly pointed out the fact that the non-plastic Bolivar fire clay did not underlie the Upper Freeport coal, but was separated from it by several distinct strata. It has been said by some that the horizon of the Bolivar clay is the same as that of the

Upper Freeport limestone, and that the flint clay, when present, replaces the limestone. It may be this is often the case, but there are exposures which distinctly show both the fire clay and the limestone present. There has been considerable confusion caused by this wrongly calling the Bolivar the Upper Freeport. It is seldom when the flint clay is present that the true Upper Freeport is not found.

I must object to the loose way in which the term "kaolin" is used. If the term "kaolin" is to be used to designate any white clay, then let us use the term "white clay" and we will know what is meant. Dr. Sellards is to be congratulated that in his last Florida report, in speaking of the white clays there, he designates them as "ball-clays," which is really what they are, and the same term should be used to designate a great many other alluvial white clays. The word "kaolin" is rapidly coming to have no fixed meaning in the ceramic industry, being daily exploited by clay producers for purely commercial purposes, where they have materials that no one would pretend to substitute in a batch for kaolin. Neither should the term "kaolin" be applied to *all residual* clays, many of which are white but which do not possess the character and qualities of kaolins, and are not formed from the decomposition of igneous materials, but from secondary rocks.

As fine grinding and washing of a true kaolin increase the plasticity of the resultant remaining material, and we still call the remaining material kaolin, it has been suggested we should call the clay material which is broken down under natural conditions and been washed by nature and not artificially, a kaolin also. The very facts are an argument against this use of the term. Very fine grinding, accompanied by washing, is artificially producing the same conditions, but not in the intensified form that nature has used in the formation of sedimentary clays from once igneous rocks. As a matter of fact the extremely fine artificially ground and washed material should not and cannot properly be called "kaolin" without a modifying adjective. If the term "kaolin" is to be applied to plastic or semi-plastic white or varicolored clays formed by sedimentary deposition of material derived from the decomposition of igneous rocks at a considerable distance, being deposited and broken up and redeposited again

a number of times, we should extend the use of the term to embrace any clay whatever, and a common ordinary red-burning brick clay, found along a stream, or a hard laminated shale, should also be termed "kaolins," because they have been deposited in exactly the same manner, and it is possible to show the many steps in the transformation of material directly derived from the decomposed igneous rocks, through semi-plastic and plastic white clays, into various colored clays, with changes in the impurities present, into and through all lower grade materials, until finally deposited as mud banks along the ocean shore, and no one can draw the line when it ceases to be "kaolin," except where it leaves the parent bed—when it first changes from a residual material into a transported one.

STATE GEOLOGIST, PENNSYLVANIA,
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H. RIES: The occurrence of nodular bodies in many refractory clays, which has been touched on by Mr. Lovejoy, is one that has been described by different observers, but the origin of which has never been explained for all. We are, of course, very familiar with this structure in bauxite, where it is almost universally found, but it is not so common in refractory clays.

Mr. Lovejoy refers to it in the white sedimentary clays of the Lower Cretaceous in Georgia. Here we find lenses of white clay in the Lower Cretaceous, which are sometimes associated with true bauxite,[1] but within the clay itself there are sometimes lenses of nodular material, which are not bauxite, but consist of a mixture of kaolinite grains and quartz.[2] Nodular bodies have also been found in some of the Pennsylvania fire clays, but so far as I know they have not been examined petrographically to determine their mineral composition.

Some years ago Mr. Greaves-Walker[3] described a peculiar flint clay from Kentucky which contained similar oölites, which

[1] H. K. Shearer, A Report on the Bauxite and Fullers Earth of the Coastal Plain of Georgia, Ga. Geol. Surv., *Bull.* 31 (1917).

[2] R. E. Somers, Microscopic Examination of Clays, *J. Wash. Acad. Sci.*, 9, No. 5, 113 (1919).

[3] A. F. Greaves-Walker, "Note on a High Alumina Flint Clay," *Trans. Am. Ceram. Soc.*, 9, 461 (1907).

were subsequently found to be made up of the mineral gibbsite.[1] Mr. Walker, I believe, suggested the name "aluminite" for this clay, but as this has already been applied to a mineral it would have no validity.

A curious example of nodular clays is that found in the flint clay district of Missouri, where the material, sometimes made up entirely of small round bodies, occurs as irregular masses in the flint-clay deposits. In this case the nodules are composed almost exclusively of the mineral diaspore.[2]

I cannot agree with Mr. Lovejoy in his use of the term "kaolin." This was originally applied to white residual clays, and its application to sedimentary ones has in my opinion been the result mainly of commercial propaganda. It should be restricted to its original use. The two classes of white clay are not alike in their characters, nor can we even say that they are used for the same purposes, except perhaps in a very few cases. A somewhat similar confusion exists regarding the terms kaolin and kaolinite. The former is a rock name, and does not represent a substance of definite chemical composition, consequently there cannot be a kaolin ratio as Mr. Lovejoy states. Kaolinite, on the other hand, is a mineral of definite composition represented by the formula, $Al_2O_3.2SiO_2.2H_2O$, and may occur in almost any kind of clay, although it is most abundant in the high-grade clays, although even here it may not be as abundant as mica.[2]

CORNELL UNIVERSITY,
ITHACA, N. Y.

H. A. WHEELER: Mr. Lovejoy's generalizations that shales are always plastic but to variable degrees, while the fire clays range from marked plasticity to non-plastic in the case of flint clays, also holds good in the Missouri deposits.

The Missouri flint clays, which occupy small, local, disconnected basins in sink holes in pre-Pennsylvanian limestones (Mississippian and Silurian) usually weather in fine, angular granules on the outcrop, but in a few instances they become plastic for a few feet from the surface.

[1] S. L. Galpin, *Trans. Am. Ceram. Soc.*, 14, 301 (1912).
[2] R. E. Somers, *Loc. cit.*

That the coloring matter in some red and pink clays is organic and hence burns out is true of a few Missouri clays, as observed by Mr. Lovejoy. In some instances, I think this reddish coloring matter will be found due to petroleum, where the oil has gradually infiltrated through the clay, in doing which more or less refining and refractionating occurs.

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Ellis Lovejoy: I have examined a number of the flint fire clay deposits in Pennsylvania but chiefly the Mercer clays, and I had very little first-hand information in regard to the Lower Kittanning flint, and so stated. I am glad to be corrected by Mr. Hice but I get some comfort from the report of the Topographic and Geologic Survey of Pennsylvania, 1906–1908, page 312, wherein I find under the heading of Lower Kittanning Clay, the statement: "In many parts of the area a valuable flint clay is associated with the plastic clay." And again on page 320 is the following: "Mention has already been made of the wide extent of flint clay associated with the plastic clay under the Lower Kittanning coal."

Relative to the Upper Freeport coal, in the Fourth Series, *Bull.* **20,** Ohio Geological Survey, is the statement that the clay is directly below the coal, and also mention is made of the concretionary matter in the clay. I do not recall ever finding the coal and clay together in the same opening in Ohio but I have found them on opposite sides of the same hill and at the same level so far as it was possible to determine. In a mine in George's Creek, Md., cutting through all the coals from Brookville to the Upper Freeport inclusive, the coal of the latter horizon rests immediately upon the clay. I am pleased to have Mr. Hice bring out the fact that the clay in Pennsylvania does not underlie the coal.

The clay, whether with the coal or separate from it, is concretionary in the several districts mentioned. Petrographically, it is alike wherever found throughout the basin, and widely different from any other clay in the same area.

The geological conditions under which the bed was developed

were the same and have given the clay a marked feature in the unusual number and size of iron carbonate concretions.

The character is so marked as to correlate the clay as the same horizon in the three states. Over the wide area represented, it would not be strange if some portions of the bed of the clay had been covered with sediments upon which the coal growth again took root, without any break in the continuity of the coal bed. Splits in coal beds are frequently found and why may we not have a split by wedged shaped masses of sediments between a coal and a clay without breaking the continuity of either the coal bed or the clay?

If the coal accompanying the clay in Ohio and Maryland is the Upper Freeport, then in my opinion the clay in Pennsylvania belongs to the Upper Freeport horizon.

I do not wish to defend my use of the term "kaolin" ratio. It was loosely used but the average clayworker understands kaolin to mean a white clay with approximately the composition of kaolinite.

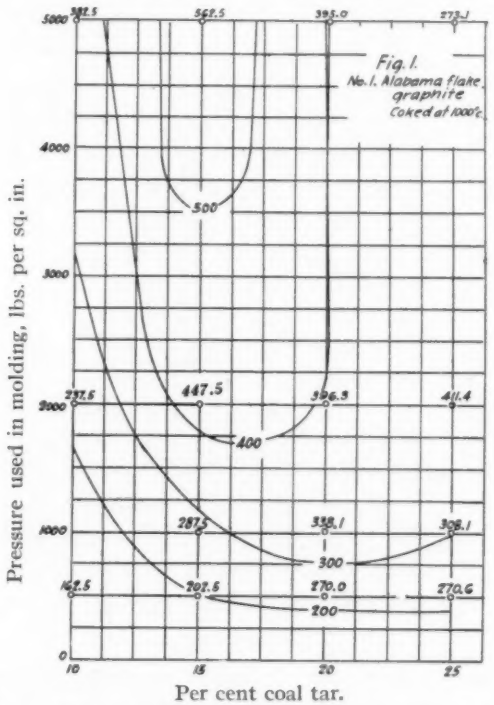
The nodules from the Georgia deposit could not have been a mixture of kaolinite and quartz, as suggested by Dr. Ries, because the silica content of such a mixture would be higher than that of kaolinite, or "kaolin ratio," as I put it.

I am of the opinion that the nodule in question was in a transition stage from kaolinite to bauxite, with none of the resulting minerals removed. Analytically, it had the composition of kaolinite, but mineralogically it was likely a mixture of a highly aluminous mineral—perhaps bauxite—and quartz, with or without undecomposed kaolinite.

EFFECT OF VARIABLE PRESSURE AND TAR CONTENT ON THE BRIQUETTING OF ALABAMA GRAPHITE.[1]

By R. T. Stull and H. G. Schurecht.

The Ceylon graphite occurs in comparatively large lumps and when ground and screened produces a material having a granular



Effect of tar and pressure on compressive strength of coked Alabama graphite.

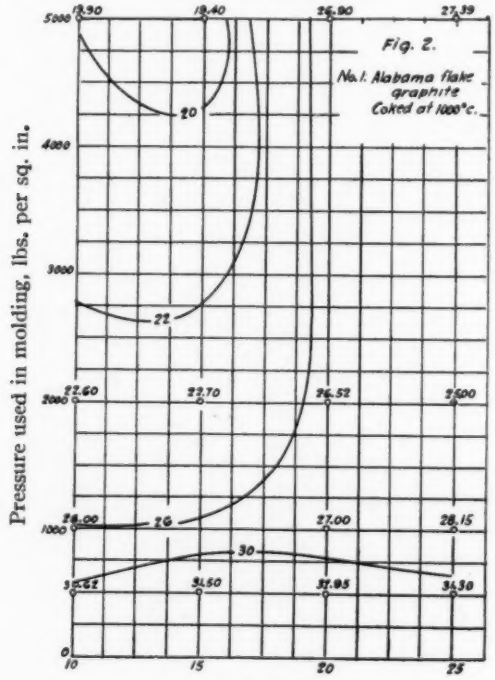
or splintery structure. On account of the comparatively large size of the lumps, it can be ground to almost any size of meshing desired. On the other hand, the Alabama graphite when pre-

[1] By permission of the Director, U. S. Bureau of Mines.

pared for the market occurs in a thin flat plate-like form seldom coarser than a 20-mesh size.

The argument which has been advanced by crucible-makers—to support their contention that the Ceylon graphite is superior to the flake form for the manufacture of crucibles—is that the granular and splintery graphite grains produce a stronger and

The effect of tar and pressure on the porosity of coked graphite.

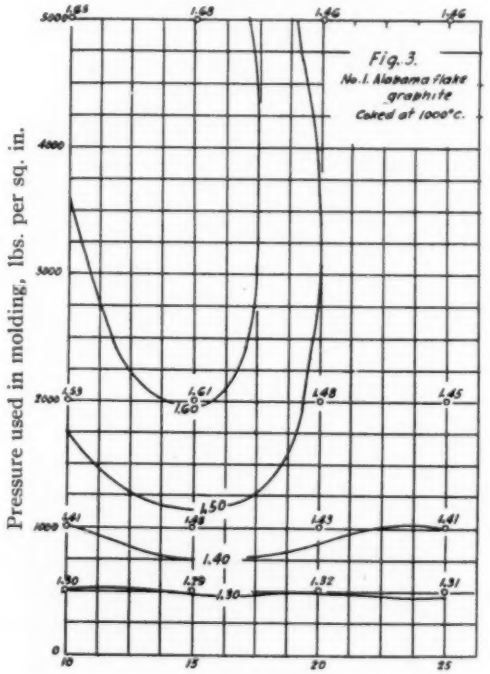


more durable crucible, due to the interlocking or "tying in" of the granules and splinters with the bond clay. On the other hand, the structure in the flake graphite body appears to be that of over-lapping plates which, it is claimed by a minority of domestic crucible makers, when bound together produce a stronger body than the granular form.

It was therefore suggested that if the Alabama flake graphite

could be modified in such a manner as to produce a granular form, larger than 20-mesh in size, the crucible making properties of graphite so prepared could be compared with those of the flake and the Ceylon, thereby throwing some light upon the question. Experiments were made to convert the Alabama flake graphite to the granular form by briquetting the graphite with

Effect of tar and pressure on the density of coked Alabama graphite.



tar, coking, crushing and screening. In order to produce a high degree of "compactness" and to approximate the Ceylon grain as closely as possible, a study of the effects of different briquetting pressures and different per cents of tar binder was undertaken.

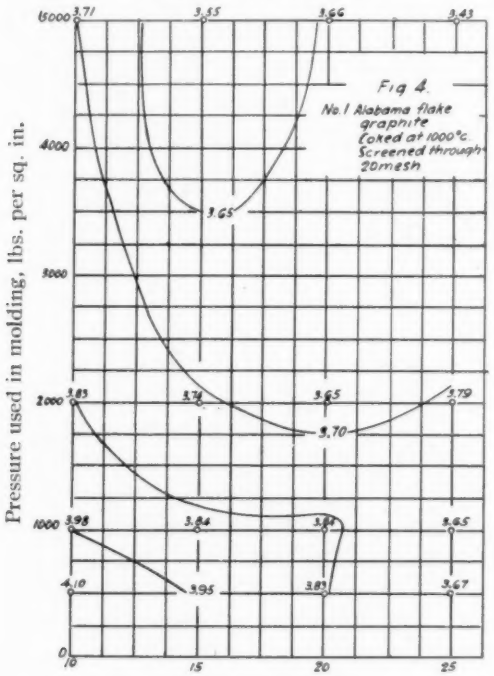
Two series were made. In Series 1 the Alabama flake graphite, screened between 20- and 100-mesh, was mixed with 10, 15, 20 and 25 percent coal tar (by weight), briquetted at different pres-

sures up to 5000 pounds per square inch on an Olsen compression testing machine, and coked rapidly at 1000° C.

Series 2 was a duplicate of Series 1 except that a mixture of three parts flake graphite and one part graphite dust passing the 100-mesh screen was used.

In order to determine the best pressure and per cent of tar to

The effect of tar and pressure on the bulkiness of Alabama coked graphite.



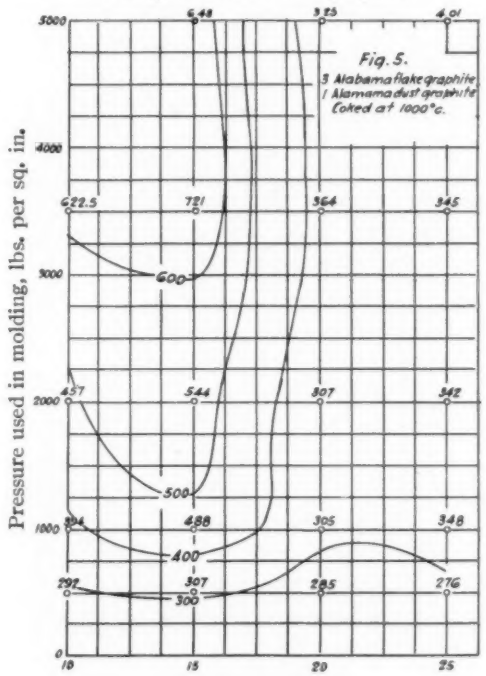
employ, the crushing strength, porosity, apparent density and bulkiness measurements were made on the coked samples.

The compressive strength tests were made on five check trials for each different set of conditions on cylinders $2^1/_2''$ in diameter and $3''$ high.

The porosity and apparent density determinations were made by the kerosene oil-immersion method on three check trials.

Bulkiness measurements were made by crushing the coked briquettes and screening between 20 and 100 mesh in order to secure results comparable with the tests on the 20-mesh flake graphite. Eighty grams of the prepared material with about 140 cc. of distilled water were placed in a graduated cylinder and the graphite allowed to settle for 24 hours, when the height

Effect of tar and pressure on compression strength of coked Ala. graphite.



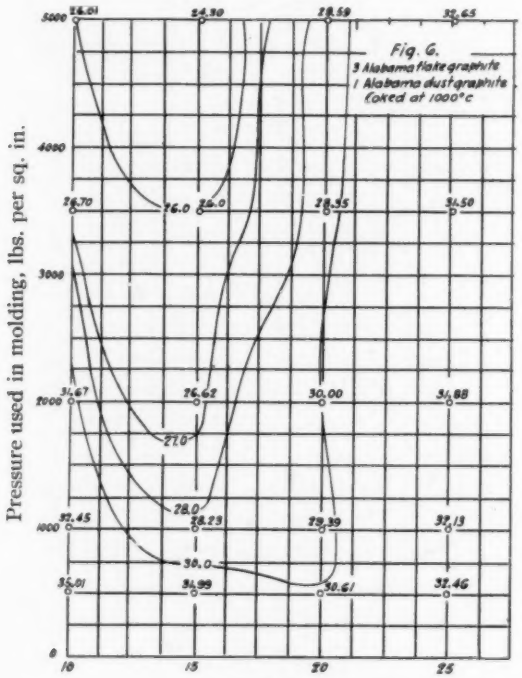
of the graphite in cubic centimeters was noted. The bulkiness was expressed in terms of the ratio of the number of cubic centimeters of graphite observed, divided by its true volume. For comparison, bulkiness measurements were also made on Ceylon, Canadian, and No. 1 Alabama flake graphites. All were screened between 20 and 100 mesh.

The comparative bulkiness was found to be as follows:

	Bulkiness factor.
Ceylon graphite	2.36
Canadian graphite	3.51
Alabama coked	3.54
Alabama flake	3.90

In order to show graphically the results obtained, crushing strength, porosity, apparent density and bulkiness lines were

Effect of tar and pressure on porosity of coked Ala. graphite.

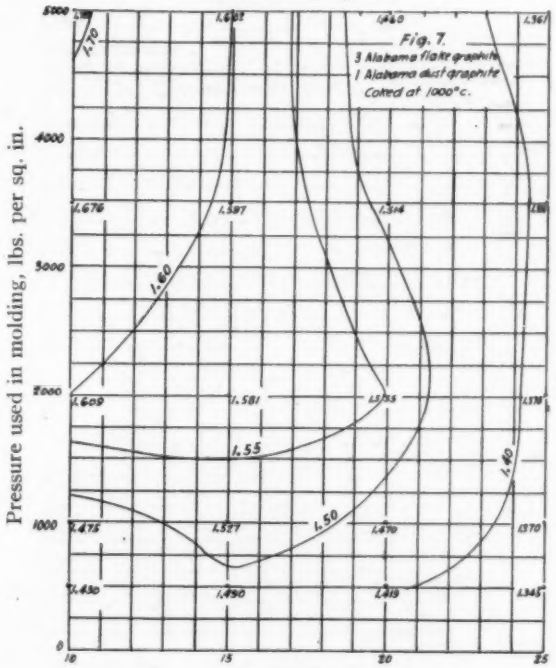


plotted. The pounds per square inch under which the briquettes were compressed are spaced on the ordinates and the percentages of tar spaced on the abscissae. Figs. 1, 2 3 and 4 show the results for Series 1, and Figs. 5, 6, 7 and 8 for Series 2.

As anticipated, the curves show that highest crushing strength, lowest porosity, highest density and lowest bulkiness occur

with highest pressures of briquetting in both series. In Series 1 for the flake graphite, the curves show that about 14 to 15 per cent tar gives the maximum crushing strength, minimum porosity, maximum density and minimum bulkiness for the highest briquetting pressures, and, in general, as the briquetting pressure decreases the amount of tar must increase up to a limit of ap-

Effect of tar and pressure on the density of coked Ala. graphite.



Per cent tar.

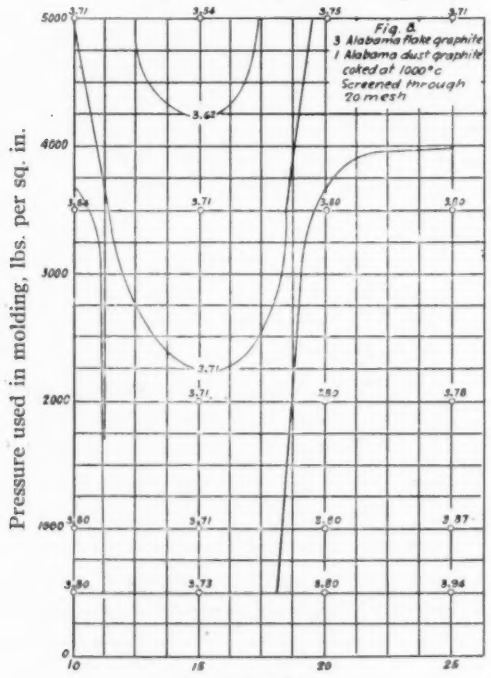
proximately 20 per cent for the lowest briquetting pressures in order to give the most desirable results.

With the mixture of flake and dust graphite, Series 2, maximum crushing strength, minimum porosity, and minimum bulkiness for each briquetting pressure occur with 15 per cent tar binder. Maximum density occurs with 10 per cent tar at 5000 pounds briquetting pressure and with 15 per cent tar at 500 pounds pressure.

High compressive strength, high density, low porosity and low bulkiness are the properties most desired. These were obtained with the highest briquetting pressure employed (5000 pounds per square inch) and for any definite addition of tar, were most nearly attained with 15 per cent tar binder.

A comparison of Series 1 and 2 at 5000 pounds briquetting

Effect of tar and pressure on the bulkiness of coked Ala. graphite.



pressure and 15 per cent tar content shows that the mixture of flake and dust gave higher compressive strength and higher porosity than the flake graphite and approximately the same density and bulkiness as the flake graphite.

Two hundred pounds of the coked graphite was prepared for making crucibles. This consisted of three parts No. 1 Alabama flake of No. 20-mesh size and one part 100-mesh graphite dust.

Fifteen per cent of coal tar by weight was selected as the binder and the briquettes were formed under a pressure of 5000 pounds per square inch. After coking at 1000° C the briquettes were crushed and screened between 16- and 100-mesh sieves.

The Alabama flake graphite has a decidedly greasy feel, while the "slippery" feeling of the Ceylon is much less. As compared to the flake and Ceylon, the coked grain appears harsh and the greasy feel is almost lacking. As compared to the Ceylon grain, the coked grain is short and "chunky," whereas the Ceylon grain is longer and narrower.

Two series of crucibles were made, one containing variable amounts of Ceylon and No. 1 Alabama flake graphite, and the other different per cents of Ceylon and coked Alabama graphite.

The crucibles containing all Alabama graphite molded with difficulty. Owing to its slipperiness, the mixture seemed to stick to the jigger tool and slide rather than pack against the mold. It did not seem to weld readily and laminated badly and when removed from the mold showed numerous welding seams. The Ceylon graphite crucibles molded much better than the flake mixture but showed a slight tendency to laminate and an occasional welding seam. The coked graphite molded perfectly, showed no tendency to laminate and no welding seams. The crucibles came from the molds apparently flawless as far as molding properties were concerned.

The crucibles were tested in a large brass foundry on two different grades of brass. Contrary to predictions, the all Alabama flake crucibles gave better service than the coked graphite crucibles, and both the coked and flake graphite crucibles gave better service than the Ceylon crucibles. The results are interesting though not conclusive—since only four crucibles of each mix were tested.[1]

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[1] For more complete data regarding tests of these crucibles see "Behavior Under Brass Foundry Practice of Crucibles Containing Ceylon, Canadian and Alabama Graphites." *J. Am. Ceram. Soc.*, **2**, 208 (March 1919).

THE PROGRESS OF VITRIFICATION AND SOLUTION IN SOME PORCELAIN MIXTURES.

By Arthur S. Watts.

Introduction.

Investigators in the field of porcelains have generally held the view that the development of desirable properties is evidenced by, first, the development of sillimanite, and second, by the prog-

Fig. 1.—Body "W," fired to cone 9.

ress of solution of the quartz grains. Both of these processes are possible of study by the use of the microscope.

In common with others, we have spent considerable time and

money in the microscopic study of porcelains but with mediocre success. Some bodies that under the microscope appear very unpromising, have proven by physical tests to be of the highest grade, while other bodies, showing great promise according to the microscopic classification, have failed completely in service. From this it would appear that we are either failing to observe

FIG. 2.—Body "W," fired to cone 11.

some vital step in the development as evidenced by the microscopic study or that certain changes are not apparent under the microscope.

Investigation.

In order to study the progress of sillimanite development and quartz solution, we prepared a body, designated "W," containing 33

per cent Canadian feldspar, 17 per cent flint, 25 per cent American china clay, and 25 per cent American ball clay. After dividing into three equal portions, one was fired to cone 9, one to cone 11, and one to cone 13. Micro-photographs of these specimens are shown in Figs. 1, 2 and 3.

A study of the specimen fired at cone 9 (Fig. 1) impresses one

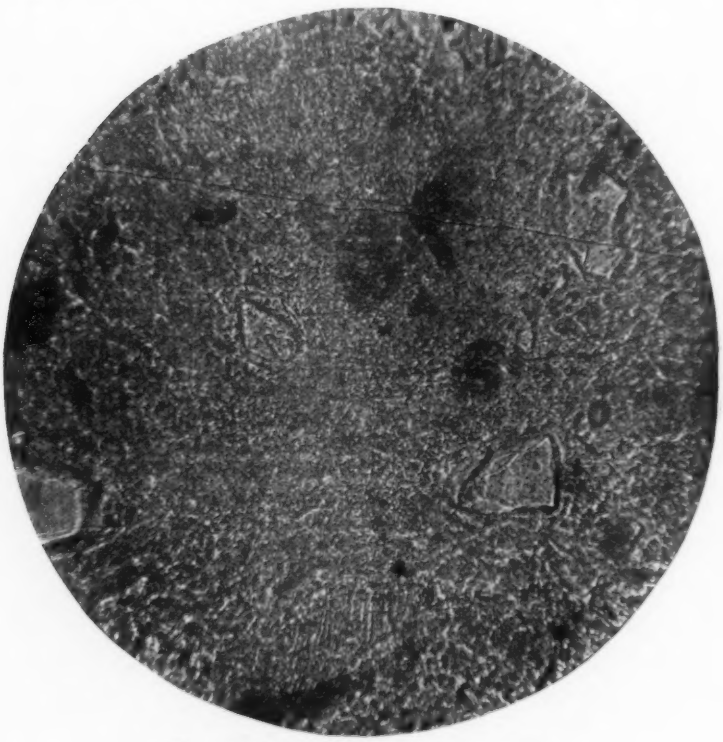

FIG. 3.—Body "W," fired to cone 13.

with the sharpness of outline of the angular quartz particles and the more or less circular particles of the larger feldspar grains, each of which is filled with fine needles of sillimanite. Other limited areas of sillimanite also appear but these are not distributed throughout the entire mass.

A study of the specimen fired at cone 11 (Fig. 2), shows the

progressive solution of the quartz particles, which have lost their corners, and the complete solution of the feldspar particles. The development of sillimanite is also much more distinct both in the number and size of crystals. It is worthy of mention that at this temperature this body attained its maximum mechanical

FIG. 4.—Body "O"

strength and had a high dielectric strength. It was also non-absorbent.

A study of the specimen fired at cone 13 (Fig. 3) indicates a distinct change in the appearance of the body. The quartz grains are completely dissolved on the edges. The remainder of the mass, however, has taken on a decidedly milky appearance and the sillimanite crystals, while they have increased in amount and appear throughout the entire matrix, are so clouded by the matrix that they might easily be overlooked. The dielectric

strength has slightly increased but the mechanical strength is slightly less than that of the same body when fired to cone 11.

In the study of this body, fired at the different temperatures, it may be worthy of note that the cavities (Figs. 1, 2 and 3) which appear as dark areas approach circular shape as the temperature of firing increases. It is also worthy of note that the

FIG. 5.—Body "L."

size of the sillimanite crystals does not increase with increase of temperature. This may be explained by the increased solubility of the clay substance in the matrix which maintains a high state of viscosity at elevated temperatures and retards crystal development. If our information regarding the solubility of quartz is true, however, the amount of quartz present should tend to overcome this viscosity by its increased solubility in the feldspar.

This raises the question of the influence of the degree of viscosity of the solvent upon the sillimanite development. In

FIG. 6.—Body "V."

studying this phase of the subject a series of bodies having the following compositions were prepared:

	"O" Body. Per cent.	"L" Body. Per cent.	"V" Body. Per cent.
Potash feldspar	21.35	21.35	...
Soda feldspar	...	...	21.35
Whiting	...	1.92	1.92
English china clay	42.93	42.93	42.93
English ball clay	8.00	8.00	8.00
Flint	26.73	26.73	26.73

These bodies were fired to cone 12 under oxidizing conditions.

Body "O" (Fig. 4), which contains no flux except potash feldspar, shows sillimanite development to about the same degree as was observed in the all-American body when fired at cone 9. The quartz grains, while smaller in size, also show about the same degree of solution as in the cone 9 body.

Body "L" (Fig. 5), which contains about 2 per cent whiting

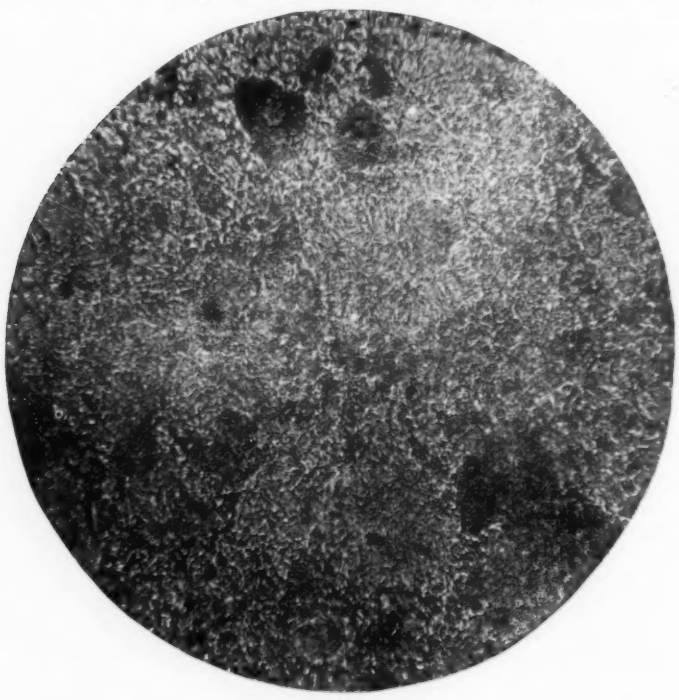

FIG. 7.—Body "Q."

as flux in addition to the potash feldspar, does not display any increased sillimanite development, but the matrix is noticeably more glossy and the quartz grains show slightly more solution.

Body "V" is exactly the same as body "L," except that soda feldspar was substituted for potash feldspar. This substitution had a marked effect both in the degree of quartz solution and sillimanite development (Fig. 6). The quartz grains have gone into solution to a degree far in excess of that attained in any

of the other bodies studied. It is noticeable also that the sillimanite crystals are at least five times as large as those in any other body studied. The distribution of sillimanite, however, is not more general than in the potash-feldspar bodies. The matrix is perhaps somewhat more glossy and numerous small spherical cavities are noted—indicating the development of a vesicular structure.

Another interesting and instructive study was made and is here presented by Body "Q" (Fig. 7). This body is of the same composition as body "L" (Fig. 5), but differs in that the feldspar, whiting, and a portion of the clay and flint were previously fused and reground thus rendering them presumably more active as fluxes. This body was fired to cone 10. The degree of solution is distinctly apparent in the quartz grains remaining and the crystals of sillimanite are more sharply defined and of slightly increased size over those in Body "L," although much smaller than those present in Body "V." The most striking improvement is in the transparency of the matrix. The presence of large numbers of tiny spherical cavities indicate that this body was over-fired even more than was the case with Body "V."

Summary.

The foregoing data indicate that the development of sillimanite starts in the feldspar grains and progresses as fusion progresses. The matrix of fused feldspar saturated with clay substance and quartz permeates the mass—the whole mass thus becoming more homogeneous.

The introduction of the calcium flux does not apparently add to the sillimanite development, although it undoubtedly increases the solution of the quartz grains at any given temperature.

The substitution of soda-feldspar for potash-feldspar is accompanied by increase in size of the sillimanite crystals but the amount of sillimanite does not apparently increase.

The preliminary fusion of the feldspar and calcium flux before incorporation in the body adds to the fluxing action and tends to increase the size of the sillimanite crystals developed. It also aids the solution of the quartz grains but the amount of sillimanite developed is not materially increased.

Conclusions.

The solution of quartz in feldspar or feldspar-calcium glass is apparently proportional to the degree of fusion and the viscosity of the mass.

The sillimanite development in a porcelain is evidently the result of a high state of saturation of an alkaline-aluminum-silicate. While the potash-feldspar porcelain shows the largest amount of sillimanite, this may be explained by the fact that the soda-feldspar used contained a larger amount of magnesia than did the potash-feldspar used. The addition of calcium undoubtedly develops a calcium-aluminum-silicate which does not encourage the development of sillimanite. It is reasonable to assume that this calcium-aluminum-silicate has a distinct solvent action on the clay substance and the natural result would be sillimanite development—if the same laws hold for all fusions saturated with clay substance. The fact that all of the clay substance is not dissolved would indicate that the fusion containing calcium has either not reached a state of saturation or that the presence of the calcium-aluminum-silicate is unfavorable to sillimanite development.

A study of natural rocks in which sillimanite and its allotropes occur would indicate that the presence of the alkaline earths is unfavorable to sillimanite development. For example, cyanite, andalusite and sillimanite, all of the composition $Al_2O_3.SiO_2$, are found in nature only in rocks of high silica content and associated with secondary minerals containing an alkali, alumina and silica. The chief of these are muscovite ($K_2O.Al_2O_3.6SiO_2.2H_2O$), paragonite ($Na_2O.2Al_2O_3.6SiO_2.H_2O$) and lepidolite ($KLiO.Al_2O_3.3SiO_2.H_2O.F$). Thus we see that the rocks containing sillimanite and its allotropes are high in alumina and alkali. Selective crystallization has enabled the alkali present to claim its maximum amount of alumina and the alumina excess enters into combination with an equal molecular amount of silica to form sillimanite or one of its allotropes.

There is no reason why this same process should not operate in the development of porcelain. The clay substance when once broken up by the fused feldspar would first saturate the feldspar and

the excess alumina would combine with an equal molecular amount of silica to form sillimanite. As in nature, we do not find sillimanite or its related minerals developing in rocks containing alkaline earth, so in the porcelain we find the presence of calcium acting in a manner unfavorable to sillimanite development.

The industrial application of porcelain requires that three questions be answered, *viz.*:

(1) How may sillimanite be developed in a porcelain so that the highest grade of product will result?

(2) To what extent can sillimanite be developed in a porcelain without the loss of any desirable physical property?

(3) To what extent can the solution of quartz progress to the benefit of the porcelain?

Some light has been thrown on the first question by this study, but much remains to be done.

The loss in mechanical strength displayed by Body "W," when fired at cone 13, indicates that while sillimanite is an important constituent, its development is not always an evidence of improved physical properties.

A simple study reported by the writer indicates that incorporation of the flint into a calcine or semi-frit before introduction into the porcelain increases its resistance to shock, but such limited research is not a safe basis for a general statement and an exhaustive study is required before any general conclusions can be safely drawn.

DEPARTMENT OF CERAMIC ENGINEERING,
OHIO STATE UNIVERSITY,
COLUMBUS, OHIO

AN APPARATUS FOR STUDYING THE DISSOCIATION OF CARBONATE ROCKS.

By. G. A. Bole.

Introduction.

It is common knowledge that a static determination of dissociation pressures not only requires complicated apparatus, but also a great amount of experience and painstaking manipulation to obtain even fairly accurate results.

The results of carefully carried out determinations of the temperature at which the pressure of CO_2 becomes atmospheric vary between 812° C and 915° C, for calcium carbonate, depending on whether the work of Le Chatelier or Zavriev be considered. The results of the work of other careful investigators, such as Johnston, Potts, and De Bray, fall somewhere between these two extremes, each claiming special accuracy for his work.

The disadvantage in depending on a lag in a time-temperature curve for dissociation temperatures is apparent, when one considers the fact that different parts of the sample vary in temperature from 50 to 100° C, and that the CO_2 liberated is in equilibrium with only that CaO which is at the lowest temperature. The more slowly the temperature is raised, the more accurate the results, however, and it can be said in its favor that the method approaches the conditions under which the ceramist works.

Description of the Apparatus.

For use in our laboratory we have devised a very simple piece of apparatus which has given satisfactory results even in the hands of student manipulators. It has been used not only for determining the temperature at which the pressure of the products of dissociation becomes atmospheric and for deriving the data for plotting dissociation curves of carbonate rocks, but also for determining the rate of dissociation of carbonates at different temperatures. It is of value for control work in calcination processes and for the analysis of magnesian limestones, where

it is required to distinguish between the CO_2 combined with the magnesia and that in union with the lime.

The apparatus is in effect a Victor Meyer vapor-density apparatus, in which the steam jacket has been replaced by an

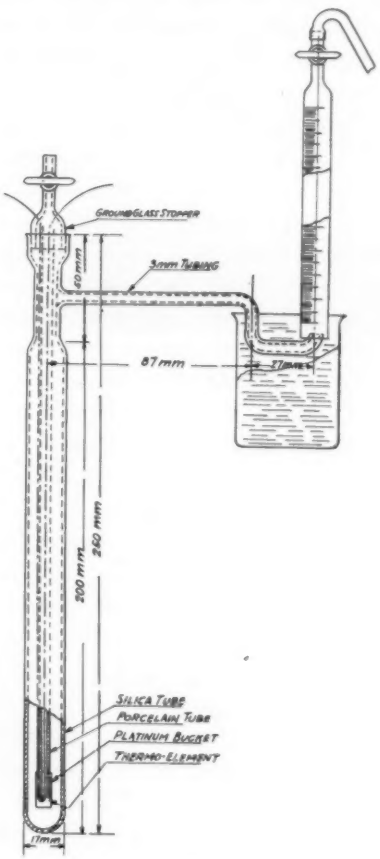

Fig. 1.

electrical tube furnace. The ends of this are well insulated to insure an even heat. The inner tube is of clear fused silica. This latter, shown in Fig. 1, is 10 inches long and $^1/_2$ inch in diameter at the larger end. Eight inches from the closed end it is somewhat

constricted in order that an asbestos disc may be introduced, to keep the hot gases from coming in contact with the rubber stopper —in case such a stopper is used instead of one of ground glass. Half way up the constricted portion a delivery tube is fused in. This is bent in such a fashion as to lead into a water- or mercury-trough. The open end of the inner tube is closed by means of a ground glass stopper, into which are fused the wires of a thermo-element, and also a tube containing a ground-glass stopcock. The gases evolved through the side delivery tube are measured over water, saturated with the products of the dissociation, or over mercury in an inverted burette which has been accurately calibrated.

As few joints as possible are desirable in order to insure against leakage. The parts of the tube outside the furnace are small, thus obviating so far as possible any expansion or contraction of the gas within the tube. Determinations should be carried out in a room of even temperature and devoid of drafts. The expansion and contraction of the silica tube itself is negligible. The whole system is tried out against 40 to 50 mm. of mercury before each determination, to guard against a faulty insertion of the stopper.

It is of importance that the tube be inserted into the furnace to the same depth each time. This is insured by making a mark on the tube, or by having a fire-clay bench built into the furnace beyond which the tube cannot enter.

The sample (0.1 gram) is contained in a small platinum bucket which just fits over the end of the porcelain insulation tube of the thermoelement, where it is held in place by means of a splinter of porcelain. This also serves to insulate the thermoelement leads from the bucket.

The temperatures are read from either a standardized millivoltmeter or from a potentiometer-galvanometer arrangement. A thermoelement is placed in the furnace also, in order that the temperature control may be more accurate. The thermoelements are standardized against authentic transition-temperatures of pure chemical substances.

Making the Determinations.

Dissociation Curve.—When securing the data for plotting a dissociation curve of a magnesian limestone, for example, the process is as follows: The sample is introduced into the quartz glass tube and is then put into the furnace. The temperature is run up to 450° C with the stopcock open, in order to drive out any moisture. It is then closed and the temperature raised through equal temperature intervals in equal time. The volume of the gas evolved is measured at the end of each time interval. The determination is continued without interruption until the dissociation has gone to completion, that point being indicated by the volume of gas evolved. The thermometer, which is kept in the water, is read after each time-interval to ascertain the temperature of the gas which has bubbled through it. The gas evolved is run through absorption pipettes, and if any carbon monoxide is found to be present, the sample is treated for removal of organic matter and a new determination is made.

The gas is evolved at atmospheric pressure, but as it ascends the tube, displacing the water, the bubbles increase in size—due to reduced pressure. Therefore, after each reading, the height of the water must be measured and subtracted from the barometric pressure, together with the vapor pressure of the water for the temperature at which the determination is being carried out. Part of the gas evolved is due to expansion of the air in the tube, and part to the products of dissociation, so it is necessary to run a blank with the platinum bucket empty, which gives the expansion curve. This curve when plotted on the same sheet as the total volume curve enables one to see the true dissociation, or the air expansion for each equal temperature interval may be subtracted from the total volume of gas evolved during that interval, and plotted thus against time.

Dissociation Temperature.—When determining the temperature at which the pressure of the products of dissociation just overcome the atmospheric (dissociation pressure) it is necessary only to find at what point the gas begins to be evolved when the temperature is held constant. The procedure is as follows: The sample and apparatus are arranged as when securing data

for a dissociation curve. The temperature is increased with the stopcock closed until a rapid evolution of gas indicates that dissociation has begun. The temperature is allowed to fall until bubbles cease to be evolved. The stopcock is then opened to equalize the pressure within the tube. If no gas comes off when the cock is again closed, the temperature has fallen below the dissociation point.

The furnace is again heated till evolution of gas sets in. It is then allowed to fall very slowly, and the stopcock opened from time to time until a point is reached where gas just ceases to come off. This temperature is held for thirty minutes and if no gas is evolved it is evident that this point lies just below the dissociation temperature. The same operation is repeated, holding the temperature five degrees higher than previously. If gas is evolved at this point it is evidently one slightly above the dissociation temperature. Carrying out this method carefully one can determine the temperature of dissociation as accurately as the temperature reading device permits, generally from 2 to 5° C.

Rate of Dissociation.—When the rate of dissociation is being determined the procedure is somewhat modified (See Fig. 2). The sample (0.5 gram) is weighed in the platinum bucket which is placed in the neck of the quartz-glass tube, being held in place by a wire which slips through the glass stopcock. Since it is impracticable to have a thermoelement inside the tube while running these determinations, the quartz tube is placed in a larger porcelain tube, in the bottom of which the thermoelement is inserted. The space between the two tubes serves as a constant temperature bath.

The arrangement is then placed in the furnace and the temperature brought up to about 50° C above the point at which the determination is to be made. It is allowed to fall slowly until the desired temperature is reached, where it is held for fifteen minutes. In this way it has been found that the temperature within and without the tube never varies more than 5° C. The bucket is now released and allowed to fall to the bottom of the tube. The stopcock is closed so that the bubbles evolved, due to dissociation, ascend in the measuring burette. There is a sudden evolu-

tion of gas, due to some of the hot gases being driven to the upper part of the tube and the colder gases descending, but there is an equal subsequent contraction as the temperature of the tube again adjusts itself to that of the furnace. The time at which the stop-cock was closed is noted, and the reaction is allowed to proceed for a

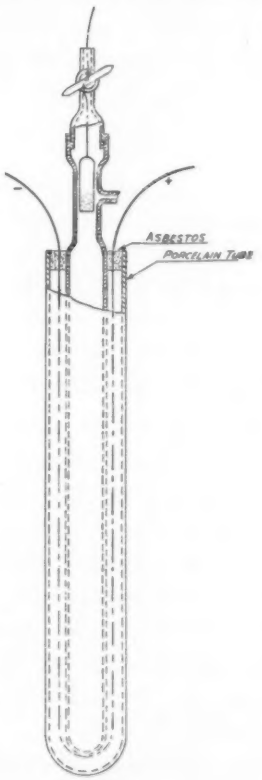

Fig. 2.

definite period, after which it is opened to the atmosphere and the volume of evoluted gas read. The bucket is withdrawn, a new sample of exactly the same weight introduced, and a determination run in the same way at a higher temperature. When sufficient points have been determined the corrected volumes are plotted against temperature.

Quantitative Analyses.—The apparatus has also been used to determine the carbon dioxide combined with the magnesia and that combined with the lime in a partially calcined dolomite. The procedure requires that the temperature be run up to 750° C with the sample suspended in the neck of the tube. When the temperature has become constant the bucket is released and the gas evolved at that temperature is measured. The temperature is raised to 900° C, where the CO_2, due to dissociation of $CaCO_3$, will be given off rapidly. The previously determined expansion, due to the rise through 150°, is subtracted from the reading at 900° C.

Carbonate rocks can be analyzed quantitatively for magnesian and lime CO_2 in the same way. MgO and CaO calculated from the CO_2 evolved at the two temperatures give results that do not vary from the average of several gravimetric determinations in a greater degree than the gravimetric determinations among themselves when fairly pure dolomites are used.

Advantages and Conclusions.

(1) A determination can be run in a short time.

(2) Impurities such as organic matter, occluded gases, and moisture can be driven off just before a determination is run, by simply manipulating a stopcock.

(3) The pressure can be equalized repeatedly during a determination by opening a stopcock. In this way metastable equilibrium and excess pressure on the apparatus are avoided.

(4) Working at the same pressure at all times (atmospheric) the inaccuracies of static measuring instruments, and substances used therewith, are avoided.

(5) The filling of the apparatus is simple, and the accuracy of the temperature is assured.

(6) The use of a transparent tube enables one to ascertain with certainty whether the thermoelement is actually in its proper position, both before and after a determination.

(7) The evolved gas can be analyzed. Thus one can be sure of the dissociation products being purely those of the reaction: $RCO_3 \longrightarrow RO + CO_2$.

The apparatus may be used for several processes:

(1) Determination of the temperature of dissociation.

(2) Determination of the rate of dissociation.

(3) Control work in calcination processes.

(4) Analyses of magnesian limestones, when it is required to distinguish between the CO_2 combined with the CaO and that combined with the MgO.

N. Y. State School of Ceramics,
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Yingling, Walter A., Supt. Woodbury Glass Co., Winchester, Ind.

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ACTIVITIES OF THE SOCIETY.

May 9th. In accordance with the recommendation of the Division of Chemistry and Chemical Technology of the National Research Council, it was voted to approve the admission of neutral countries to the International Chemical Council.

May 9th. The University Bank, Alfred, New York, was approved as the depository for the funds of the Society by the Secretary each month.

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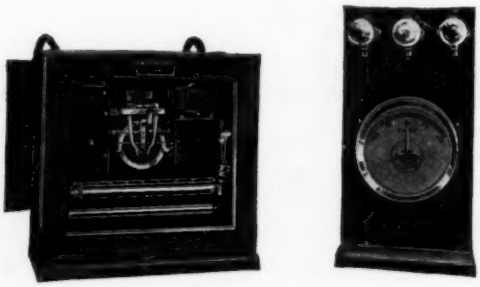

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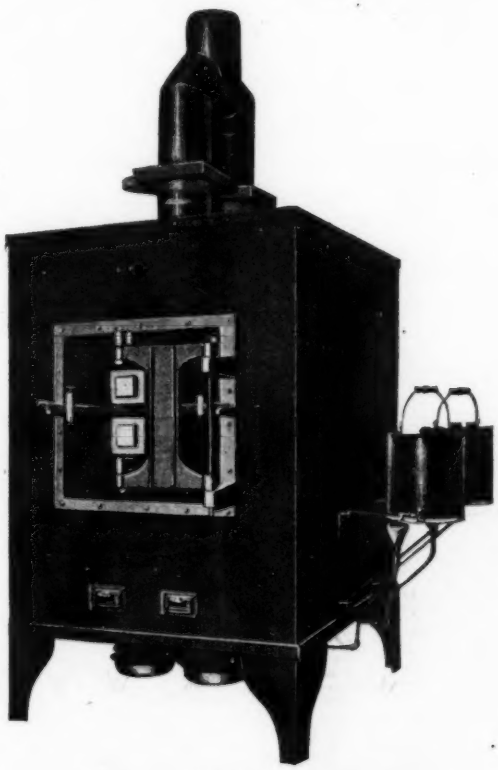

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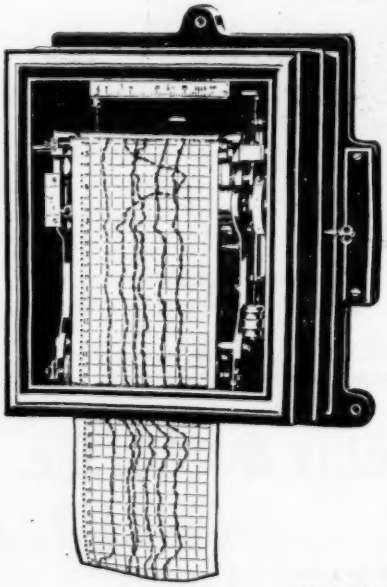

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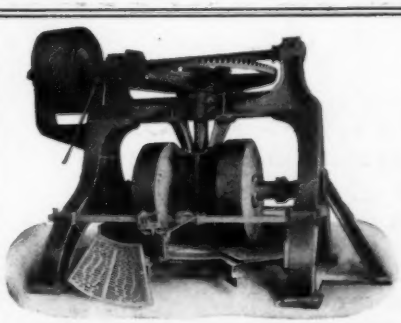

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